

SEARCH REQUEST FORM

Scientific and Technical Information Center

Requester's Full Name: BEN JACKAY Examiner #: 73489 Date: 10/20/02
Art Unit: 1620 Phone Number 305-8889 Serial Number: 10/253,297
Mail Box and Bldg/Room Location: UMI 3E11 Results Format Preferred (circle): PAPER DISK E-MAIL

If more than one search is submitted, please prioritize searches in order of need.

Please provide a detailed statement of the search topic, and describe as specifically as possible the subject matter to be searched.
Include the elected species or structures, keywords, synonyms, acronyms, and registry numbers, and combine with the concept or utility of the invention. Define any terms that may have a special meaning. Give examples or relevant citations, authors, etc, if known. Please attach a copy of the cover sheet, pertinent claims, and abstract.

Title of Invention: Process for making vitamin E using Hydrogen-tris(oxalato)phosphate

Inventors (please provide full names): BONRAH et al.

Earliest Priority Filing Date: _____

For Sequence Searches Only Please include all pertinent information (parent, child, divisional, or issued patent numbers) along with the appropriate serial number.

Process for making (all-rac)- α -tocopherol comprising a reaction mixture comprising trimethylhydroquinone and isophytol or phytol with a catalyst comprising hydrogen tris(oxalato)phosphate and an organic solvent.

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DEC 31 2002
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Online Time: 16

Type of Search

NA Sequence (#) _____ STN ✓
AA Sequence (#) _____ Dialog _____
Structure (#) 2 Questel/Orbit _____
Bibliographic _____ Dr.Link _____
Litigation _____ Lexis/Nexis ✓
Fulltext _____ Sequence Systems _____
Patent Family _____ WWW/Internet _____
Other _____ Other (specify) _____

Vendors and cost where applicable

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IFIUDB			
NEWS	6	Apr 22	Records from IP.com available in CAPLUS, HCAPLUS, and
ZCAPLUS			
NEWS	7	Apr 22	BIOSIS Gene Names now available in TOXCENTER
NEWS	8	Apr 22	Federal Research in Progress (FEDRIP) now available
NEWS	9	Jun 03	New e-mail delivery for search results now available
NEWS	10	Jun 10	MEDLINE Reload
NEWS	11	Jun 10	PCTFULL has been reloaded
NEWS	12	Jul 02	FOREGE no longer contains STANDARDS file segment
NEWS	13	Jul 22	USAN to be reloaded July 28, 2002;
			saved answer sets no longer valid
NEWS	14	Jul 29	Enhanced polymer searching in REGISTRY
NEWS	15	Jul 30	NETFIRST to be removed from STN
NEWS	16	Aug 08	CANCERLIT reload
NEWS	17	Aug 08	PHARMAMarketLetter(PHARMAML) - new on STN
NEWS	18	Aug 08	NTIS has been reloaded and enhanced
NEWS	19	Aug 19	Aquatic Toxicity Information Retrieval (AQUIRE)
			now available on STN
NEWS	20	Aug 19	IFIPAT, IFICDB, and IFIUDB have been reloaded
NEWS	21	Aug 19	The MEDLINE file segment of TOXCENTER has been reloaded
NEWS	22	Aug 26	Sequence searching in REGISTRY enhanced
NEWS	23	Sep 03	JAPIO has been reloaded and enhanced
NEWS	24	Sep 16	Experimental properties added to the REGISTRY file
NEWS	25	Sep 16	CA Section Thesaurus available in CAPLUS and CA
NEWS	26	Oct 01	CASREACT Enriched with Reactions from 1907 to 1985
NEWS	27	Oct 21	EVENTLINE has been reloaded
NEWS	28	Oct 24	BEILSTEIN adds new search fields
NEWS	29	Oct 24	Nutraceuticals International (NUTRACEUT) now available on
STN			
NEWS	30	Oct 25	MEDLINE SDI run of October 8, 2002
NEWS	31	Nov 18	DKILIT has been renamed APOLLIT
NEWS	32	Nov 25	More calculated properties added to REGISTRY
NEWS	33	Dec 02	TIBKAT will be removed from STN
NEWS	34	Dec 04	CSA files on STN
NEWS	35	Dec 17	PCTFULL now covers WP/PCT Applications from 1978 to date
NEWS	36	Dec 17	TOXCENTER enhanced with additional content
NEWS	37	Dec 17	Adis Clinical Trials Insight now available on STN
NEWS	38	Dec 30	ISMEC no longer available

NEWS 39 Jan 13 Indexing added to some pre-1967 records in CA/CAPLUS
NEWS 40 Jan 21 NUTRACEUT offering one free connect hour in February 2003
NEWS 41 Jan 21 PHARMAML offering one free connect hour in February 2003
NEWS 42 Jan 29 Simultaneous left and right truncation added to COMPENDEX,
ENERGY, INSPEC
NEWS 43 Feb 13 CANCERLIT is no longer being updated
NEWS 44 Feb 24 METADEX enhancements
NEWS 45 Feb 24 PCTGEN now available on STN
NEWS 46 Feb 24 TEMA now available on STN

NEWS EXPRESS January 6 CURRENT WINDOWS VERSION IS V6.01a,
CURRENT MACINTOSH VERSION IS V6.0b(ENG) AND V6.0Jb(JP),
AND CURRENT DISCOVER FILE IS DATED 01 OCTOBER 2002
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* * * * * STN Columbus * * * * *

FILE 'HOME' ENTERED AT 18:06:25 ON 25 FEB 2003

=> file reg

COST IN U.S. DOLLARS

SINCE FILE	TOTAL
ENTRY	SESSION
0.21	0.21

FULL ESTIMATED COST

FILE 'REGISTRY' ENTERED AT 18:06:34 ON 25 FEB 2003

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Property values tagged with IC are from the ZIC/VINITI data file
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STRUCTURE FILE UPDATES: 24 FEB 2003 HIGHEST RN 494745-03-8

DICTIONARY FILE UPDATES: 24 FEB 2003 HIGHEST RN 494745-03-8

TSCA INFORMATION NOW CURRENT THROUGH MAY 20, 2002

Please note that search-term pricing does apply when
conducting SmartSELECT searches.

Crossover limits have been increased. See HELP CROSSOVER for details.

Experimental and calculated property data are now available. See HELP PROPERTIES for more information. See STNote 27, Searching Properties in the CAS Registry File, for complete details:
<http://www.cas.org/ONLINE/STN/STNOTES/stnotes27.pdf>

```
=> s 174749-62-3/rn
L1          1 174749-62-3/RN
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```
=> d l1 all
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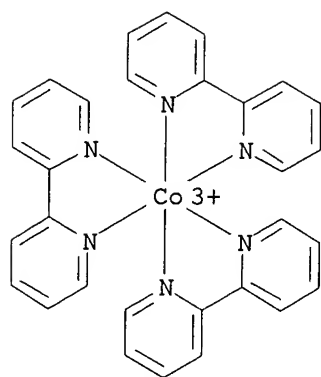
```
L1  ANSWER 1 OF 1  REGISTRY  COPYRIGHT 2003 ACS
RN  174749-62-3  REGISTRY
CN  Cobalt(3+), tris(2,2'-bipyridine-N,N')-, (OC-6-11)-, sodium
    hexafluorophosphate(1-) (OC-6-11)-tris[ethanedioato(2-)-O,O']chromate(3-)
    (1:1:1:1) (9CI)  (CA INDEX NAME)
OTHER CA INDEX NAMES:
CN  Chromate(3-), tris[ethanedioato(2-)-O,O']-, (OC-6-11)-, sodium
    (OC-6-11)-tris(2,2'-bipyridine-N,N')cobalt(3+) hexafluorophosphate(1-)
    (1:1:1:1) (9CI)
OTHER NAMES:
CN  Tris(2,2'-bipyridine)cobalt(3+) tris(oxalato)chromate(3-) sodium
    hexafluorophosphate(1-)
MF  C30 H24 Co N6 . C6 Cr O12 . F6 P . Na
SR  CA
LC  STN Files:  CA, CAPLUS
```

Ring System Data

Elemental Analysis EA	Elemental Sequence ES	Size of the Rings SZ	Ring System Formula RF	Ring Identifier RID	RID Occurrence Count
C2CrO2-	CrOC2O-	5-5-5	C6CrO6	1523.80.1	1 in CM
C2CrO2-C2CrO2	CrOC2O-CrOC2O				3
C2CoN2-	CoNC2N-	5-5-5-6-6-6	C30CoN6	13130.7.1	1 in CM
C2CoN2-	CoNC2N-	6-6-6			1
C2CoN2-C5N-	CoNC2N-NC5-				
C5N-C5N-C5N-	NC5-NC5-NC5-				
C5N-C5N	NC5-NC5				

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CM  1
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CRN  19052-39-2
CMF  C30 H24 Co N6
CCI  CCS
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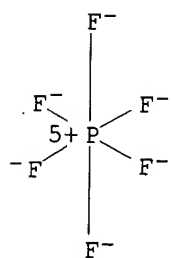


CM 2

CRN 16919-18-9

CMF F6 P

CCI CCS

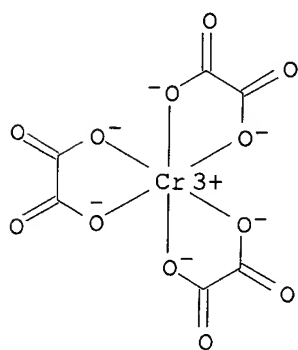


CM 3

CRN 15054-01-0

CMF C6 Cr O12

CCI CCS



1 REFERENCES IN FILE CA (1962 TO DATE)
 1 REFERENCES IN FILE CAPLUS (1962 TO DATE)

REFERENCE 1

- AN 124:248655 CA
 TI Chiral, Three-Dimensional Supramolecular Compounds: Homo- and Bimetallic Oxalate- and 1,2-Dithiooxalate-Bridged Networks. A Structural and Photophysical Study
 AU Decurtins, Silvio; Schmalle, Helmut W.; Pellaux, Rene; Schneuwly, Philippe; Hauser, Andreas
 CS Institut fuer Anorganische Chemie, Universitaet Zuerich, Zurich, CH-8057, Switz.
 SO Inorganic Chemistry (1996), 35(6), 1451-60
 CODEN: INOCAJ; ISSN: 0020-1669
 PB American Chemical Society
 DT Journal
 LA English
 CC 78-7 (Inorganic Chemicals and Reactions)
 Section cross-reference(s): 73, 75
 AB In analogy to the [MII(bpy)3]2+ cations (MII = transition-metal; bpy = 2,2'-bipyridine), [MIII(bpy)3]3+ cations (MIII = Cr or Co) induce the crystn. of chiral, anionic three-dimensional (3D) coordination polymers of oxalate-bridged (μ -ox) metal complexes [MII2(ox)3]n2n- or [MIMIII(ox)3]n2n-. The tripos. charge is partially compensated by inclusion of addnl. complex anions like ClO4-, BF4-, or PF6- which are encapsulated in cubic shaped cavities formed by the bipyridine ligands of the cations. Thus, an elaborate structure of cationic and anionic species within a polymeric anionic network is realized. The compds. isolated and structurally characterized include [Cr(bpy)3][ClO4] [NaCr(ox)3] (1), [Cr(bpy)3][ClO4][Mn2(ox)3] (2), [Cr(bpy)3][BF4][Mn2(ox)3] (3), [Co(bpy)3][PF6][NaCr(ox)3] (4). Crystal data: 1, cubic, space group P213, a 15.523(4) .ANG., Z = 4; 2, cubic, space group P4132, a 15.564(3) .ANG., Z = 4; 3, cubic, P4132, a 15.553(3) .ANG., Z = 4; 4, cubic, P213, a 15.515(3) .ANG., Z = 4. Also, it seemed likely that 1,2-dithiooxalate (dto) could act as an alternative to the oxalate bridging ligand, and as a result [Ni(phen)3][NaCo(dto)3].cntdot.C3H6O (5) has successfully been isolated and structurally characterized. Crystal data: 5, orthorhombic, space group P212121, a 16.238(4), b 16.225(4), c 18.371(5) .ANG., Z = 4. The photophys. properties of 1 were studied. In single crystal absorption spectra of [Cr(bpy)3][ClO4][NaCr(ox)3] (1), the spin-flip transitions of both the [Cr(bpy)3]3+ and the [Cr(ox)3]3- chromophores are obsd. and can be clearly distinguished. Irradiating into the spin-allowed 4A2 \rightarrow 4T2 absorption band of [Cr(ox)3]3- results in intense luminescence from the 2E state of [Cr(bpy)3]3+ as a result of rapid energy transfer processes.
 ST crystal structure chromium cobalt bipyridine oxalatometalate; bipyridine chromium cobalt oxalatometalate prepn; manganate oxalato chromium cobalt bipyridine prepn; thiooxalato cobaltate nickel phenanthroline prepn structure; photophys chromium bipyridine oxalatochromate; luminescence chromium bipyridine oxalatochromate; absorption spectra chromium

bipyridine oxalatochromate
 IT Energy level transition
 Energy transfer
 Luminescence
 Ultraviolet and visible spectra
 (of bipyridinechromium oxalatochromate)
 IT Crystal structure
 Molecular structure
 (of chromium/cobalt bipyridine oxalatochromate and chromium
 phenanthroline dithiooxalatochromate complexes)
 IT Imines
 RL: PRP (Properties); SPN (Synthetic preparation); PREP (Preparation)
 (di-, transition metal complexes, oxalatochromate/manganate and
 dithiooxalatocobaltate; prepn. and crystal structure of)
 IT Transition metal compounds
 RL: PRP (Properties); SPN (Synthetic preparation); PREP (Preparation)
 (diimine complexes, oxalatochromate/manganate and
 dithiooxalatocobaltate; prepn. and crystal structure of)
 IT 23539-86-8, Tris(2,2'-bipyridine)chromium(3+) triperchlorate
 RL: RCT (Reactant); RACT (Reactant or reagent)
 (for prepn. of chromium bipyridine oxalatochromate/manganate
 complexes)
 IT 174749-67-8, Tris(2,2'-bipyridine)chromium(3+)
 tris(tetrafluoroborate(1-))
 RL: RCT (Reactant); RACT (Reactant or reagent)
 (for prepn. of chromium bipyridine oxalatomanganate complexes)
 IT 14217-01-7, Tripotassium tris(oxalato)chromate(3-)
 RL: RCT (Reactant); RACT (Reactant or reagent)
 (for prepn. of chromium/cobalt bipyridine oxalatochromate complexes)
 IT 28277-53-4, Tris(2,2'-bipyridine)cobalt(3+) tris(hexafluorophosphate(1-))
 RL: RCT (Reactant); RACT (Reactant or reagent)
 (for prepn. of cobalt bipyridine oxalatochromate complex)
 IT 39016-57-4, Tris(1,10-phenanthroline)nickel(2+) sulfate 92348-93-1
 RL: RCT (Reactant); RACT (Reactant or reagent)
 (for prepn. of nickel phenanthroline dithiooxalatocobaltate complex)
 IT 174749-59-8P, Tris(2,2'-bipyridine)chromium(3+) tris(oxalato)chromate(3-)
 sodium perchlorate
 RL: PEP (Physical, engineering or chemical process); PRP (Properties);
 SPN
 (Synthetic preparation); PREP (Preparation); PROC (Process)
 (prepn. and crystal structure and photophys. of)
 IT 174749-60-1P, Tris(2,2'-bipyridine)chromium(3+)
 tris(oxalato)dimanganate(2-)
) perchlorate 174749-62-3P, Tris(2,2'-bipyridine)cobalt(3+)
 tris(oxalato)chromate(3-) sodium hexafluorophosphate(1-) 174749-64-5P,
 Tris(1,10-phenanthroline)nickel(2+) tris(dithiooxalato)cobaltate(3-)
 sodium acetone
 RL: PRP (Properties); SPN (Synthetic preparation); PREP (Preparation)
 (prepn. and crystal structure of)
 IT 174749-59-8DP, Tris(2,2'-bipyridine)chromium(3+)
 tris(oxalato)chromate(3-)
 sodium perchlorate, solid soln. with tris(bipyridine)rhodium analog
 174749-66-7DP, Tris(2,2'-bipyridine)rhodium(3+) tris(oxalato)chromate(3-)
 sodium perchlorate, solid soln. with tris(bipyridine)chromium analog
 RL: PEP (Physical, engineering or chemical process); PRP (Properties);
 SPN

(Synthetic preparation); PREP (Preparation); PROC (Process)
(prepn. and luminescence of)

IT 174749-61-2P

RL: PRP (Properties); SPN (Synthetic preparation); PREP (Preparation)
(s.c.9; codpn. and crystal structure of)

IT 174749-61-2P

RL: PRP (Properties); SPN (Synthetic preparation); PREP (Preparation)
(s.c.9; prepn. and crystal structure of)

=> file caplus

COST IN U.S. DOLLARS

SINCE FILE	TOTAL
ENTRY	SESSION
7.83	8.04

FULL ESTIMATED COST

DISCOUNT AMOUNTS (FOR QUALIFYING ACCOUNTS)

SINCE FILE	TOTAL
ENTRY	SESSION
-0.62	-0.62

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FILE COVERS 1907 - 25 Feb 2003 VOL 138 ISS 9

FILE LAST UPDATED: 24 Feb 2003 (20030224/ED)

This file contains CAS Registry Numbers for easy and accurate substance identification.

=> s l1 and chelate

1 L1

39189 CHELATE

L2

0 L1 AND CHELATE

=> s l1 and catalyst

1 L1

614401 CATALYST

L3

0 L1 AND CATALYST

=> s l1 and organic synthesis

1 L1

286892 ORGANIC

1044916 SYNTHESIS

7826 ORGANIC SYNTHESIS

(ORGANIC(W)SYNTHESIS)

L4 0 L1 AND ORGANIC SYNTHESIS

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	ENTRY	SESSION
FULL ESTIMATED COST	7.72	15.76
DISCOUNT AMOUNTS (FOR QUALIFYING ACCOUNTS)	SINCE FILE	TOTAL
	ENTRY	SESSION
CA SUBSCRIBER PRICE	0.00	-0.62

STN INTERNATIONAL LOGOFF AT 18:12:00 ON 25 FEB 2003

=> s ?tocopherol? (L) ?hydroquinon? (l) ?phytol? (l) phospha?
26251 ?TOCOPHEROL?
48390 ?HYDROQUINON?
3511 ?PHYTOL?
696077 PHOSPHA?
L1 4 ?TOCOPHEROL? (L) ?HYDROQUINON? (L) ?PHYTOL? (L) PHOSPHA?

=> d bib hit 1-4

L1 ANSWER 1 OF 4 CAPLUS COPYRIGHT 2003 ACS
AN 2002:930649 CAPLUS
TI Final Report on the Safety Assessment of Tocopherol, Tocopheryl Acetate, Tocopheryl Linoleate, Tocopheryl Linoleate/Oleate, Tocopheryl Nicotinate, Tocopheryl Succinate, Dioleoyl Tocopheryl Methylsilanol, Potassium Ascorbyl Tocopheryl Phosphate, and Tocophersolan
AU Fiume, Monice Zondlo
CS NW, 1101 17th Street, Cosmetic Ingredient Review, Suite 310, Washington, DC, 20036, USA
SO International Journal of Toxicology (2002), 21(6), 51-116
CODEN: IJTOfN; ISSN: 1091-5818
PB Taylor & Francis Ltd.
DT Journal
LA English
AB **Tocopherol** and its several ester and ether derivs. all function as antioxidants in cosmetic formulations; they also have other functions, such as skin conditioning. Tocopheryl Acetate, **Tocopherol**, and Tocopheryl Linoleate are used in 2673 formulations, generally at concns. of up to 36, 5, and 2, resp., although Tocopheryl Acetate is 100 of vitamin E oil. Tocophersolan, Tocopheryl Linoleate/Oleate, Tocopheryl Nicotinate, Tocopheryl Succinate, Dioleoyl Tocopheryl Methylsilanol, and Potassium Ascorbyl Tocopheryl **Phosphate**, combined, are used in 36 formulations at concns. lower than those reported for the frequently used ingredients. **Tocopherol** may be isolated from vegetable oils or synthesized using **isophytol** and **methylhydroquinone**. **Tocopherol**, Tocopheryl Acetate, Tocopheryl Linoleate, and Tocopheryl Succinate all were absorbed in human skin. In rat skin, Tocopheryl Acetate is hydrolyzed to **Tocopherol**. **Tocopherol** is a natural component of cell membranes thought to protect against oxidative damage. **Tocopherol**, Tocopheryl Acetate, and Tocopheryl Succinate each were reported to protect against UV radiation- induced skin damage. These ingredients are generally not toxic in animal feeding studies, although very high doses (.gtoreq.2 g/kg/day) have hemorrhagic activity. These ingredients are generally not irritating or sensitizing to skin or irritating to eyes, although a Tocopheryl Acetate did produce sensitization in one animal test, and Tocophersolan was a slight eye irritant in an animal test. Reproductive and developmental toxicity tests in animals using **Tocopherol**, Tocopheryl Acetate, Tocopheryl Succinate, and Tocophersolan were all neg. or showed some effect of reducing toxicity. **Tocopherol**, Tocopheryl Acetate, Tocopheryl Succinate, and Dioleoyl Tocopheryl Methylsilanol were almost uniformly neg. These ingredients exhibit antimutagenic activity consistent with their antioxidant properties. **Tocopherol** was not carcinogenic. The ability of **Tocopherol**, Tocopheryl Acetate, and Tocopheryl Succinate to modulate the carcinogenic effect of other agents (e.g., tumor promotion) has been extensively studied. One study showing tumor promotion in mice may be discounted as not reproducible and not consistent with the large vol. of data suggesting that the antioxidant properties of these agents protect against tumor induction. Specifically, the frequent use of **Tocopherol** as a neg. control in other tumor promotion studies suggests that **Tocopherol** is not a tumor promoter. **Tocopherol** has been shown to reduce the photocarcinogenic effect

of UV radiation in mice. Similar studies with Tocopheryl Acetate and Tocopheryl Succinate, however, demonstrated some enhancement of photocarcinogenesis, although the effect was not dose related. In clin. studies, **Tocopherol**, Tocopheryl Acetate, and Tocopheryl Nicotinate were not irritants or sensitizers. A report of a large no. of pos. patch-tests to Tocopheryl Linoleate in one cosmetic product were considered to result from a contaminant or metabolite. The Cosmetic Ingredient Review Expert Panel considered that these data provide an adequate basis on which to conclude that **Tocopherol**, Tocophersolan, Tocopheryl Acetate, Tocopheryl Linoleate, Tocopheryl Linoleate/Oleate, Tocopheryl Nicotinate, Tocopheryl Succinate, Dioleoyl Tocopheryl Methylsilanol, and Potassium Ascorbyl Tocopheryl **Phosphate** are safe as used in cosmetic formulations. Although there were no inhalation toxicity data, these ingredients are used at such low concns. in hair sprays that no inhalation toxicity risk was considered likely. Because **methylhydroquinone** is used in the chem. synthesis of **Tocopherol**, there was concern that **hydroquinone** may be present as an impurity. In such cases, residual levels of **hydroquinone** would be expected to be limited to those achieved by good manufg. practices.

L1 ANSWER 2 OF 4 CAPLUS COPYRIGHT 2003 ACS
 AN 2002:573361 CAPLUS.
 DN 137:125303
 TI Manufacture of (all-rac)-.alpha.-tocopherol
 IN Bonrath, Werner; Netscher, Thomas; Wietelmann, Ulrich
 PA Roche Vitamins A.-G., Switz.
 SO Eur. Pat. Appl., 11 pp.
 CODEN: EPXXDW
 DT Patent
 LA English
 FAN.CNT 1

	PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
PI	EP 1227089	A1	20020731	EP 2002-764	20020114
	R: AT, BE, CH, DE, DK, ES, FR, GB, GR, IT, LI, LU, NL, SE, MC, PT, IE, SI, LT, LV, FI, RO, MK, CY, AL, TR				
	JP 2002284776	A2	20021003	JP 2002-5681	20020115
	CN 1365977	A	20020828	CN 2002-102027	20020117
	US 2002161247	A1	20021031	US 2002-53297	20020117
PRAI	EP 2001-101026	A	20010118		

OS CASREACT 137:125303

RE.CNT 3 THERE ARE 3 CITED REFERENCES AVAILABLE FOR THIS RECORD
 ALL CITATIONS AVAILABLE IN THE RE FORMAT

AB A process for the manuf. of (.+-.).alpha.-tocopherol by the catalyzed reaction of **trimethylhydroquinone** with **isophytol** or **phytol** is characterized by carrying out the reaction in the presence of hydrogen tris(oxalato)**phosphate**, or an adduct thereof with a solvent, as the catalyst in an org. solvent. The product of the process is the most active and industrially most important member of the vitamin E group.

L1 ANSWER 3 OF 4 CAPLUS COPYRIGHT 2003 ACS
 AN 1968:510116 CAPLUS
 DN 69:110116
 TI Monolayers of some biologically important quinones
 AU Gaines, George L., Jr.
 CS Gen. Elec. Res. and Develop. Center, Schenectady, NY, USA
 SO Journal of Colloid and Interface Science (1968), 28(2), 331-3
 CODEN: JCISA5; ISSN: 0021-9797
 DT Journal
 LA English
 AB Monolayers of ubiquinone 6 (I) and .alpha.-tocopherolquinone

(II) were spread from C₆H₆ solns. on 10-3M phosphate buffer (pH 8) solns. in dim light or darkness. The surface pressure-area curves of I and II are compared with those of vitamin K₁ and **phytol**. The area requirement in the monolayers is very nearly the same for vitamin K₁ and **phytol**; the substitution of the bulky naphthoquinone ring for a single hydroxyl group has little effect on the mol. area in these expanded films. In II, the presence of both the quinone and hydroxyl function in each mol. nearly doubles the area occupied, as well as making the film more stable to collapse. The surface potentials due to **phytol**, vitamin K₁, and II are similar, rising from .apprx.200 to .apprx.250 mv. on compression. I has an unsatd. chain, half again as long as the chain of the other compds. This longer chain, at low surface pressure, permits a greater area per mol. in the expanded film. On compression, however, the I monolayer can be compressed to nearly as small areas as vitamin K₁. The surface potential of a I film ranges from .apprx.350 to >400 mv. on compression. Attempts to demonstrate the redn. of the quinones to the corresponding **hydroquinones** in the films on aq. subphases were unsuccessful.

L1 ANSWER 4 OF 4 CAPLUS COPYRIGHT 2003 ACS

AN 1959:105575 CAPLUS

DN 53:105575

OREF 53:18965f-i,18966a

TI .alpha.-Tocopherol esters

PA E. Merck Chemische Fabrik.

DT Patent

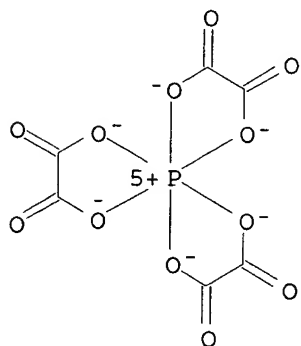
LA Unavailable

FAN.CNT 1

	PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
PI	GB 811895		19590415	GB	
AB	<p>The diethylaminoacetate of .alpha.-tocopherol was prepd. as follows: Moist 4,2,3,6-HOME3C₆H₄N₂Cl (200 g.) was added to 300 g. ClCH₂CO₂H in 600 ml. of C₆H₆ during 2 hrs. with heating and stirring. The C₆H₆ and excess ClCH₂CO₂H were removed by distn. at 60 mm. and the residue taken up in Et₂O was washed with 1% NH₃ until neutral, dried, filtered and the solvent removed by distn. The residue was stirred with low-boiling petr. ether and the crystals of trimethylhydroquinone which result were filtered off. After evapn. of the mother liquor, distn. of the residue at 1.5 mm. of Hg gave 2,5,6-trimethylhydroquinone chloroacetate (I), m. 116-17.degree.. To 48 g. I in 200 ml. C₆H₆, 30 g. phytol or isophytol and 30 g. ZnCl₂ were added. The mixt. was heated and stirred 5 hrs. in a CO₂ atm. The soln. was washed with H₂O, bleached, and the solvent removed. To sep. unreacted chloroacetate, 60 ml. petr. ether was added. Removal of the solvent and distn. at 219-30.degree. 0.01 mm. gave .alpha.-tocopherol chloroacetate (II) as a greenish yellow oil which crystd. on standing. A mixt. of II, 15 ml. Et₂NH, and 2.5 g. Na₂CO₃ was refluxed 3 hrs., an addnl. 10 ml. of amine was added and refluxing continued 2 hrs. Et₂O was added, excess NaHCO₃ sepd. by filtration and excess amine removed by washing with aq. AcOH. Evapn. of the solvent gave .alpha.-tocopherol diethylaminoacetate (III), a reddish yellow oil. II was also prepd. by refluxing a mixt. of 50 g. .alpha.-tocopherol in 200 ml. toluene to which was added 50 g. ClCH₂CO₂H during 12 hrs. in a CO₂ atm. III (5 g.) in 3 ml. abs. EtOH made acid with 2N HCl in alc. gave III.HCl, m. 156-7.degree.. Also prepd. were: III.HBr m. 170-1.degree.; III tartrate, m. 170.degree. (decompn.); .alpha.-tocopherol dimethylaminoacetate-HCl (IV.HCl), m. 192-3.degree.; IV.HBr, m. 202-3.5.degree.; IV sulfate, sinters 225.degree., decomp. 240.degree.. Prepn. of III citrate, III phosphate, and III sulfate were also reported but no phys. consts. given.</p>				

need to get reference

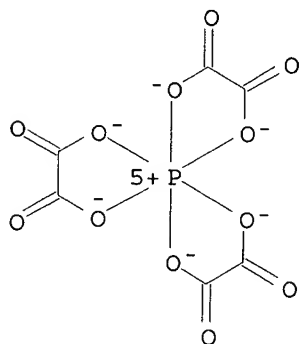
L7 ANSWER 1 OF 4 REGISTRY COPYRIGHT 2003 ACS
 RN 321201-35-8 REGISTRY
 CN Phosphate(1-), tris[ethanedioato(2-)-.kappa.O1,.kappa.O2]-, sodium,
 (OC-6-11)- (9CI) (CA INDEX NAME)
 OTHER NAMES:
 CN **Sodium tris(oxalato)phosphate(1-)**
 MF C6 O12 P . Na
 CI CCS
 SR CA
 LC STN Files: CA, CAPLUS



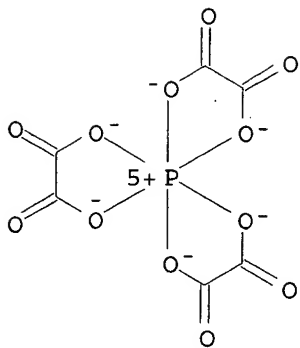
● Na⁺

1 REFERENCES IN FILE CA (1962 TO DATE)
 1 REFERENCES IN FILE CAPLUS (1962 TO DATE)

L7 ANSWER 2 OF 4 REGISTRY COPYRIGHT 2003 ACS
 RN 321201-34-7 REGISTRY
 CN Phosphate(1-), tris[ethanedioato(2-)-.kappa.O1,.kappa.O2]-, hydrogen,
 (OC-6-11)- (9CI) (CA INDEX NAME)
 OTHER NAMES:
 CN **Hydrogen tris(oxalato)phosphate(1-)**
 MF C6 O12 P . H
 CI CCS
 SR CA
 LC STN Files: CA, CAPLUS, CASREACT, USPATFULL



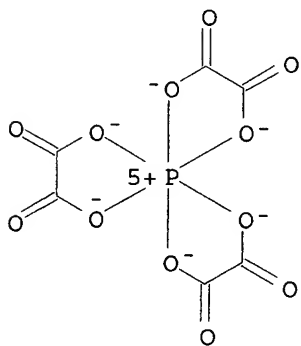
● H⁺



● H⁺

2 REFERENCES IN FILE CA (1962 TO DATE)
2 REFERENCES IN FILE CAPLUS (1962 TO DATE)

L7 ANSWER 3 OF 4 REGISTRY COPYRIGHT 2003 ACS
RN 321201-33-6 REGISTRY
CN Phosphate(1-), tris[ethanedioato(2-)-.kappa.O1,.kappa.O2]-, lithium,
(OC-6-11)- (9CI) (CA INDEX NAME)
OTHER NAMES:
CN **Lithium tris(oxalato)phosphate(1-)**
MF C6 O12 P . Li
CI CCS
SR CA
LC STN Files: CA, CAPLUS



● Li⁺

1 REFERENCES IN FILE CA (1962 TO DATE)
1 REFERENCES IN FILE CAPLUS (1962 TO DATE)

L7 ANSWER 4 OF 4 REGISTRY COPYRIGHT 2003 ACS
RN 174749-62-3 REGISTRY
CN Cobalt(3+), tris(2,2'-bipyridine-N,N')-, (OC-6-11)-, sodium
hexafluorophosphate(1-) (OC-6-11)-tris[ethanedioato(2-)-O,O']chromate(3-)
(1:1:1:1) (9CI) (CA INDEX NAME)
OTHER CA INDEX NAMES:
CN Chromate(3-), tris[ethanedioato(2-)-O,O']-, (OC-6-11)-, sodium
(OC-6-11)-tris(2,2'-bipyridine-N,N')cobalt(3+) hexafluorophosphate(1-)
(1:1:1:1) (9CI)

OTHER NAMES:

CN Tris(2,2'-bipyridine)cobalt(3+) tris(oxalato)chromate(3-) sodium hexafluorophosphate(1-)

MF C30 H24 Co N6 . C6 Cr O12 . F6 P . Na

SR CA

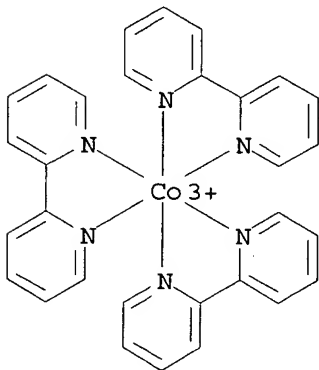
LC STN Files: CA, CAPLUS

CM 1

CRN 19052-39-2

CMF C30 H24 Co N6

CCI CCS

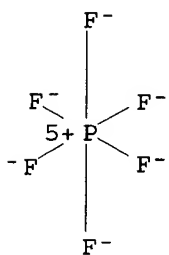


CM 2

CRN 16919-18-9

CMF F6 P

CCI CCS



CM 3

CRN 15054-01-0

CMF C6 Cr O12

CCI CCS

Crossover limits have been increased. See HELP CROSSOVER for details.

Experimental and calculated property data are now available. See HELP PROPERTIES for more information. See STNote 27, Searching Properties in the CAS Registry File, for complete details:
<http://www.cas.org/ONLINE/STN/STNOTES/stnotes27.pdf>

=> s 174749-62-3/rn

L1 1 174749-62-3/RN

=> d l1 all

L1 ANSWER 1 OF 1 REGISTRY COPYRIGHT 2003 ACS

RN 174749-62-3 REGISTRY

CN Cobalt(3+), tris(2,2'-bipyridine-N,N')-, (OC-6-11)-, sodium hexafluorophosphate(1-) (OC-6-11)-tris[ethanedioato(2-)-O,O']chromate(3-) (1:1:1:1) (9CI) (CA INDEX NAME)

OTHER CA INDEX NAMES:

CN Chromate(3-), tris[ethanedioato(2-)-O,O']-, (OC-6-11)-, sodium (OC-6-11)-tris(2,2'-bipyridine-N,N')cobalt(3+) hexafluorophosphate(1-) (1:1:1:1) (9CI)

OTHER NAMES:

CN Tris(2,2'-bipyridine)cobalt(3+) tris(oxalato)chromate(3-) sodium hexafluorophosphate(1-)

MF C30 H24 Co N6 . C6 Cr O12 . F6 P . Na

SR CA

LC STN Files: CA, CAPLUS

Ring System Data

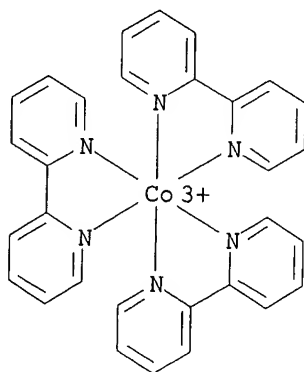
Elemental Analysis EA	Elemental Sequence ES	Size of the Rings SZ	Ring System Formula RF	Ring Identifier RID	RID Occurrence Count
C2CrO2-	CrOC2O-	5-5-5	C6CrO6	1523.80.1	1 in CM
C2CrO2-C2CrO2	CrOC2O-CrOC2O				3
C2CoN2-	CoNC2N-	5-5-5-6-6-6	C30CoN6	13130.7.1	1 in CM
C2CoN2-	CoNC2N-	6-6-6			1
C2CoN2-C5N-	CoNC2N-NC5-				
C5N-C5N-C5N-	NC5-NC5-NC5-				
C5N-C5N	NC5-NC5				

CM 1

CRN 19052-39-2

CMF C30 H24 Co N6

CCI CCS

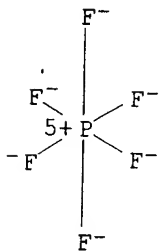


CM 2

CRN 16919-18-9

CMF F6 P

CCI CCS

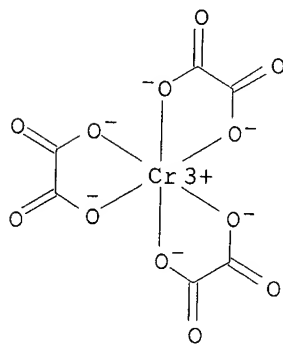


CM 3

CRN 15054-01-0

CMF C6 Cr O12

CCI CCS



bipyridine oxalatochromate

IT Energy level transition
Energy transfer
Luminescence
Ultraviolet and visible spectra
(of bipyridinechromium oxalatochromate)

IT Crystal structure
Molecular structure
(of chromium/cobalt bipyridine oxalatotransition metalate and chromium phenanthroline dithiooxalatochromate complexes)

IT Imines
RL: PRP (Properties); SPN (Synthetic preparation); PREP (Preparation)
(di-, transition metal complexes, oxalatochromate/manganate and dithiooxalatocobaltate; prepn. and crystal structure of)

IT Transition metal compounds
RL: PRP (Properties); SPN (Synthetic preparation); PREP (Preparation)
(diimine complexes, oxalatochromate/manganate and dithiooxalatocobaltate; prepn. and crystal structure of)

IT 23539-86-8, Tris(2,2'-bipyridine)chromium(3+) triperchlorate
RL: RCT (Reactant); RACT (Reactant or reagent)
(for prepn. of chromium bipyridine oxalatochromate/manganate complexes)

IT 174749-67-8, Tris(2,2'-bipyridine)chromium(3+) tris(tetrafluoroborate(1-))
RL: RCT (Reactant); RACT (Reactant or reagent)
(for prepn. of chromium bipyridine oxalatomanganate complexes)

IT 14217-01-7, Tripotassium tris(oxalato)chromate(3-)
RL: RCT (Reactant); RACT (Reactant or reagent)
(for prepn. of chromium/cobalt bipyridine oxalatochromate complexes)

IT 28277-53-4, Tris(2,2'-bipyridine)cobalt(3+) tris(hexafluorophosphate(1-))
RL: RCT (Reactant); RACT (Reactant or reagent)
(for prepn. of cobalt bipyridine oxalatochromate complex)

IT 39016-57-4, Tris(1,10-phenanthroline)nickel(2+) sulfate 92348-93-1
RL: RCT (Reactant); RACT (Reactant or reagent)
(for prepn. of nickel phenanthroline dithiooxalatocobaltate complex)

IT 174749-59-8P, Tris(2,2'-bipyridine)chromium(3+) tris(oxalato)chromate(3-) sodium perchlorate
RL: PEP (Physical, engineering or chemical process); PRP (Properties); SPN (Synthetic preparation); PREP (Preparation); PROC (Process)
(prepn. and crystal structure and photophys. of)

IT 174749-60-1P, Tris(2,2'-bipyridine)chromium(3+) tris(oxalato)dimanganate(2-)) perchlorate 174749-62-3P, Tris(2,2'-bipyridine)cobalt(3+) tris(oxalato)chromate(3-) sodium hexafluorophosphate(1-) 174749-64-5P, Tris(1,10-phenanthroline)nickel(2+) tris(dithiooxalato)cobaltate(3-) sodium acetone
RL: PRP (Properties); SPN (Synthetic preparation); PREP (Preparation)
(prepn. and crystal structure of)

IT 174749-59-8DP, Tris(2,2'-bipyridine)chromium(3+) tris(oxalato)chromate(3-) sodium perchlorate, solid soln. with tris(bipyridine)rhodium analog 174749-66-7DP, Tris(2,2'-bipyridine)rhodium(3+) tris(oxalato)chromate(3-) sodium perchlorate, solid soln. with tris(bipyridine)chromium analog
RL: PEP (Physical, engineering or chemical process); PRP (Properties); SPN

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FILE COVERS 1907 - 5 Mar 2003 VOL 138 ISS 10
FILE LAST UPDATED: 4 Mar 2003 (20030304/ED)

This file contains CAS Registry Numbers for easy and accurate substance identification.

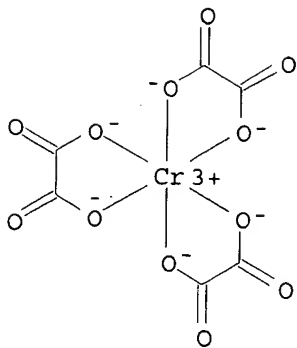
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L1 1 DE19933898/PN

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L1 ANSWER 1 OF 1 CAPLUS COPYRIGHT 2003 ACS
AN 2001:78395 CAPLUS
DN 134:125211
TI Tris(oxalato)phosphates, method for their preparation and their use
IN Wietelmann, Ulrich; Schade, Klaus; Lischka, Uwe
PA Chemetall G.m.b.H., Germany
SO PCT Int. Appl., 29 pp.
CODEN: PIXXD2
DT Patent
LA German
IC ICM C07F009-6571
ICS H01M010-40
CC 78-7 (Inorganic Chemicals and Reactions)
Section cross-reference(s): 72
FAN.CNT 1

	PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
PI	WO 2001007450	A1	20010201	WO 2000-EP4301	20000512
	W: CA, CN, JP, KR, US				
	RW: AT, BE, CH, CY, DE, DK, ES, FI, FR, GB, GR, IE, IT, LU, MC, NL, PT, SE				
	DE 19933898	A1	20010201	DE 1999-19933898	19990722 <--
	EP 1203001	A1	20020508	EP 2000-925279	20000512
	R: AT, BE, CH, DE, DK, ES, FR, GB, GR, IT, LI, LU, NL, SE, MC, PT, IE, FI, CY				
	JP 2003505464	T2	20030212	JP 2001-512534	20000512
PRAI	DE 1999-19933898	A	19990722		
	WO 2000-EP4301	W	20000512		
AB	The invention relates to tris(oxalato)phosphates, M[P(C2O4)3] (M = H, metal, N(R1R2R3R4), R1, R2, R3, R4 = independently H, C1-8 alkyl group). The invention also relates to a method for prepg. such compds. as well as to their use.				
ST	tris oxalato phosphate prepn				
IT	Quaternary ammonium compounds, preparation				
	RL: SPN (Synthetic preparation); PREP (Preparation) (prepn. of tris(oxalato)phosphate salts with)				
IT	Phosphates, preparation				
	RL: SPN (Synthetic preparation); PREP (Preparation) (tris(oxalato)phosphates; prepn. of)				
IT	321201-34-7P, Hydrogen tris(oxalato)phosphate(1-)				
	RL: RCT (Reactant); SPN (Synthetic preparation); PREP (Preparation); RACT				

(Reactant or reagent)
(prepn. and reaction with butyllithium)
IT 321201-35-8P, Sodium tris(oxalato)phosphate(1-) 321201-36-9P
321201-37-0P 321201-38-1P 321201-39-2P
RL: SPN (Synthetic preparation); PREP (Preparation)
(prepn. of)
IT 321201-33-6P, Lithium tris(oxalato)phosphate(1-)
RL: SPN (Synthetic preparation); PREP (Preparation)
(prepn. of electrolyte soln. of)
IT 10026-13-8, Phosphorus pentachloride
RL: RCT (Reactant); RACT (Reactant or reagent)
(reaction with oxalic acid)
IT 144-62-7, Oxalic acid, reactions
RL: RCT (Reactant); RACT (Reactant or reagent)
(reaction with phosphorus pentachloride)
RE.CNT 3 THERE ARE 3 CITED REFERENCES AVAILABLE FOR THIS RECORD
RE
(1) Handa, M; ELECTROCHEMICAL AND SOLID-STATE LETTERS 1999, V2(2), P60 CAPLUS
(2) Hitachi Maxell Ltd; EP 0631340 A 1994 CAPLUS
(3) Merck Patent GmbH; WO 9807729 A 1998 CAPLUS



1 REFERENCES IN FILE CA (1962 TO DATE)
1 REFERENCES IN FILE CAPLUS (1962 TO DATE)

=> filc aplus

FILC IS NOT A RECOGNIZED COMMAND

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For a list of commands available to you in the current file, enter
"HELP COMMANDS" at an arrow prompt (=>).

=> fil caplus

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	ENTRY	SESSION
FULL ESTIMATED COST	46.30	70.31
DISCOUNT AMOUNTS (FOR QUALIFYING ACCOUNTS)	SINCE FILE	TOTAL
	ENTRY	SESSION
CA SUBSCRIBER PRICE	0.00	-2.60

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FILE COVERS 1907 - 27 Jan 2003 VOL 138 ISS 5

FILE LAST UPDATED: 26 Jan 2003 (20030126/ED)

This file contains CAS Registry Numbers for easy and accurate substance identification.

=> s l7

L8 3 L7

=> s l8 and tocopherol?

26227 TOCOPHEROL?

L9 1 L8 AND TOCOPHEROL?

=> d bib abs

L9 ANSWER 1 OF 1 CAPLUS COPYRIGHT 2003 ACS
AN 2002:573361 CAPLUS
DN 137:125303
TI Manufacture of (all-rac)-.alpha.-tocopherol
IN Bonrath, Werner; Netscher, Thomas; Wietelmann, Ulrich
PA Roche Vitamins A.-G., Switz.
SO Eur. Pat. Appl., 11 pp.
CODEN: EPXXDW

DT Patent
LA English

FAN.CNT 1

	PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
PI	EP 1227089	A1	20020731	EP 2002-764	20020114
	R: AT, BE, CH, DE, DK, ES, FR, GB, GR, IT, LI, LU, NL, SE, MC, PT, IE, SI, LT, LV, FI, RO, MK, CY, AL, TR				
	JP 2002284776	A2	20021003	JP 2002-5681	20020115
	CN 1365977	A	20020828	CN 2002-102027	20020117
	US 2002161247	A1	20021031	US 2002-53297	20020117
PRAI	EP 2001-101026	A	20010118		

OS CASREACT 137:125303

AB A process for the manuf. of (.+-.)-.alpha.-tocopherol by the catalyzed reaction of trimethylhydroquinone with isophytol or phytol is characterized by carrying out the reaction in the presence of hydrogen tris(oxalato)phosphate, or an adduct thereof with a solvent, as the catalyst in an org. solvent. The product of the process is the most active and industrially most important member of the vitamin E group.

RE.CNT 3 THERE ARE 3 CITED REFERENCES AVAILABLE FOR THIS RECORD
ALL CITATIONS AVAILABLE IN THE RE FORMAT

=> d bib abs 1-3 18

L8 ANSWER 1 OF 3 CAPLUS COPYRIGHT 2003 ACS
AN 2002:573361 CAPLUS
DN 137:125303
TI Manufacture of (all-rac)-.alpha.-tocopherol
IN Bonrath, Werner; Netscher, Thomas; Wietelmann, Ulrich
PA Roche Vitamins A.-G., Switz.
SO Eur. Pat. Appl., 11 pp.
CODEN: EPXXDW

DT Patent
LA English

FAN.CNT 1

	PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
PI	EP 1227089	A1	20020731	EP 2002-764	20020114
	R: AT, BE, CH, DE, DK, ES, FR, GB, GR, IT, LI, LU, NL, SE, MC, PT, IE, SI, LT, LV, FI, RO, MK, CY, AL, TR				
	JP 2002284776	A2	20021003	JP 2002-5681	20020115
	CN 1365977	A	20020828	CN 2002-102027	20020117
	US 2002161247	A1	20021031	US 2002-53297	20020117
PRAI	EP 2001-101026	A	20010118		

OS CASREACT 137:125303

AB A process for the manuf. of (.+-.)-.alpha.-tocopherol by the catalyzed reaction of trimethylhydroquinone with isophytol or phytol is characterized by carrying out the reaction in the presence of hydrogen tris(oxalato)phosphate, or an adduct thereof with a solvent, as the catalyst in an org. solvent. The product of the process is the most active and industrially most important member of the vitamin E group.

RE.CNT 3 THERE ARE 3 CITED REFERENCES AVAILABLE FOR THIS RECORD

ALL CITATIONS AVAILABLE IN THE RE FORMAT

L8 ANSWER 2 OF 3 CAPLUS COPYRIGHT 2003 ACS
 AN 2001:78395 CAPLUS
 DN 134:125211
 TI Tris(oxalato)phosphates, method for their preparation and their use
 IN Wietelmann, Ulrich; Schade, Klaus; Lischka, Uwe
 PA Chemetall G.m.b.H., Germany
 SO PCT Int. Appl., 29 pp.
 CODEN: PIXXD2

DT Patent
 LA German
 FAN.CNT 1

2001 Date - See equivalents for potential b2(e)

	PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
PI	WO 2001007450	A1	20010201	WO 2000-EP4301	20000512
	W: CA, CN, JP, KR, US				
	RW: AT, BE, CH, CY, DE, DK, ES, FI, FR, GB, GR, IE, IT, LU, MC, NL, PT, SE				
→	DE 19933898	A1	20010201	DE 1999-19933898	19990722
→	EP 1203001	A1	20020508	EP 2000-925279	20000512
	R: AT, BE, CH, DE, DK, ES, FR, GB, GR, IT, LI, LU, NL, SE, MC, PT, IE, FI, CY				
PRAI	DE 1999-19933898	A	19990722		
	WO 2000-EP4301	W	20000512		
AB	The invention relates to tris(oxalato)phosphates, $M[P(C_2O_4)_3]$ ($M = H$, metal, $N(R_1R_2R_3R_4)$, $R_1, R_2, R_3, R_4 =$ independently H , C_1-8 alkyl group). The invention also relates to a method for prep. such compds. as well as to their use.				

RE.CNT 3 THERE ARE 3 CITED REFERENCES AVAILABLE FOR THIS RECORD
 ALL CITATIONS AVAILABLE IN THE RE FORMAT

L8 ANSWER 3 OF 3 CAPLUS COPYRIGHT 2003 ACS
 AN 1996:121444 CAPLUS
 DN 124:248655
 TI Chiral, Three-Dimensional Supramolecular Compounds: Homo- and Bimetallic Oxalate- and 1,2-Dithiooxalate-Bridged Networks. A Structural and Photophysical Study
 AU Decurtins, Silvio; Schmalle, Helmut W.; Pellaux, Rene; Schneuwly, Philippe; Hauser, Andreas
 CS Institut fuer Anorganische Chemie, Universitaet Zuerich, Zurich, CH-8057, Switz.
 SO Inorganic Chemistry (1996), 35(6), 1451-60
 CODEN: INOCAJ; ISSN: 0020-1669
 PB American Chemical Society
 DT Journal
 LA English
 AB In analogy to the $[MII(bpy)_3]^{2+}$ cations ($MII =$ transition-metal; $bpy = 2,2'$ -bipyridine), $[MIII(bpy)_3]^{3+}$ cations ($MIII = Cr$ or Co) induce the crystn. of chiral, anionic three-dimensional (3D) coordination polymers of oxalate-bridged (μ_3 -ox) metal complexes $[MII_2(ox)_3]^{n2n-}$ or $[MIMIII(ox)_3]^{n2n-}$. The tripos. charge is partially compensated by inclusion of addnl. complex anions like ClO_4^- , BF_4^- , or PF_6^- which are encapsulated in cubic shaped cavities formed by the bipyridine ligands of the cations. Thus, an elaborate structure of cationic and anionic species within a polymeric anionic network is realized. The compds. isolated and structurally characterized include $[Cr(bpy)_3][ClO_4]$ $[NaCr(ox)_3]$ (1), $[Cr(bpy)_3][ClO_4][Mn_2(ox)_3]$ (2), $[Cr(bpy)_3][BF_4][Mn_2(ox)_3]$ (3), $[Co(bpy)_3][PF_6][NaCr(ox)_3]$ (4). Crystal data: 1, cubic, space group $P213$, a 15.523(4) .ANG., $Z = 4$; 2, cubic, space group $P4132$, a 15.564(3) .ANG., $Z = 4$; 3, cubic, $P4132$, a 15.553(3) .ANG., $Z = 4$; 4, cubic, $P213$, a 15.515(3) .ANG., $Z = 4$. Also, it seemed likely that 1,2-dithiooxalate (dto) could act as an alternative to the oxalate bridging ligand, and as a

result $[\text{Ni}(\text{phen})_3][\text{NaCo}(\text{dto})_3] \cdot \text{C}_3\text{H}_6\text{O}$ (5) has successfully been isolated and structurally characterized. Crystal data: 5, orthorhombic, space group $P2_12_12_1$, a 16.238(4), b 16.225(4), c 18.371(5) Å, $Z = 4$. The photophys. properties of 1 were studied. In single crystal absorption spectra of $[\text{Cr}(\text{bpy})_3][\text{ClO}_4][\text{NaCr}(\text{ox})_3]$ (1), the spin-flip transitions of both the $[\text{Cr}(\text{bpy})_3]^{3+}$ and the $[\text{Cr}(\text{ox})_3]^{3-}$ chromophores are obsd. and can be clearly distinguished. Irradiating into the spin-allowed $4A_2 \rightarrow 4T_2$ absorption band of $[\text{Cr}(\text{ox})_3]^{3-}$ results in intense luminescence from the $2E$ state of $[\text{Cr}(\text{bpy})_3]^{3+}$ as a result of rapid energy transfer processes.

=> file reg

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TSCA INFORMATION NOW CURRENT THROUGH MAY 20, 2002

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Experimental and calculated property data are now available. See HELP PROPERTIES for more information. See STNote 27, Searching Properties in the CAS Registry File, for complete details:
<http://www.cas.org/ONLINE/STN/STNOTES/stnotes27.pdf>

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FILE COVERS 1907 - 21 Jan 2003 VOL 138 ISS 4
FILE LAST UPDATED: 20 Jan 2003 (20030120/ED)

This file contains CAS Registry Numbers for easy and accurate substance identification.

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L12 22 SEA FILE=REGISTRY ABB=ON (10026-13-8/BI OR 10191-41-0/BI OR 105-58-8/BI OR 108-10-1/BI OR 108-32-7/BI OR 108-88-3/BI OR 110-54-3/BI OR 111-65-9/BI OR 120-92-3/BI OR 1330-20-7/BI OR 142-82-5/BI OR 144-62-7/BI OR 321201-34-7/BI OR 60046-87-9/BI OR 616-38-6/BI OR 700-13-0/BI OR 71-43-2/BI OR 7541-49-3/BI OR 78-59-1/BI OR 96-22-0/BI OR 96-48-0/BI OR 96-49-1/BI)
L13 1 SEA FILE=REGISTRY ABB=ON 10191-41-0 - *α tocopherol*
L14 1032 SEA FILE=HCAPLUS ABB=ON L13
L15 104 SEA FILE=HCAPLUS ABB=ON L14(L) (PREP OR IMF OR SPN)/RL
L16 2 SEA FILE=REGISTRY ABB=ON L12 AND 1/P
L17 1 SEA FILE=REGISTRY ABB=ON L16 AND PHOSPHATE? *Catalyst*

KATHLEEN FULLER EIC 1700/PARKER LAW 308-4290

L18 2 SEA FILE=HCAPLUS ABB=ON L17
L19 1 SEA FILE=HCAPLUS ABB=ON L15 AND L18

=> d l19 all hitstr

L19 ANSWER 1 OF 1 HCAPLUS COPYRIGHT 2003 ACS
AN 2002:573361 HCAPLUS
DN 137:125303
TI Manufacture of (all-rac)-.alpha.-tocopherol
IN Bonrath, Werner; Netscher, Thomas; Wietelmann, Ulrich
PA Roche Vitamins A.-G., Switz.
SO Eur. Pat. Appl., 11 pp.
CODEN: EPXXDW
DT Patent
LA English
IC ICM C07D311-72
CC 30-20 (Terpenes and Terpenoids)
Section cross-reference(s): 29, 67

applicante

FAN.CNT 1

	PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
PI	EP 1227089	A1	20020731	EP 2002-764	20020114
	R: AT, BE, CH, DE, DK, ES, FR, GB, GR, IT, LI, LU, NL, SE, MC, PT, IE, SI, LT, LV, FI, RO, MK, CY, AL, TR				
	JP 2002284776	A2	20021003	JP 2002-5681	20020115
	CN 1365977	A	20020828	CN 2002-102027	20020117
	US 2002161247	A1	20021031	US 2002-53297	20020117
PRAI	EP 2001-101026	A	20010118		
OS	CASREACT 137:125303				
AB	A process for the manuf. of (.+-.)-.alpha.-tocopherol by the catalyzed reaction of trimethylhydroquinone with isophytol or phytol is characterized by carrying out the reaction in the presence of hydrogen tris(oxalato)phosphate, or an adduct thereof with a solvent, as the catalyst in an org. solvent. The product of the process is the most active and industrially most important member of the vitamin E group.				
ST	tocopherol prepn hydrogen trisoxalatophosphate catalyst				
IT	321201-34-7P, Hydrogen tris(oxalato)phosphate(1-) RL: CAT (Catalyst use); IMF (Industrial manufacture); SPN (Synthetic preparation); PREP (Preparation); USES (Uses) (process for the prepn. of .alpha.-tocopherol)				
IT	10191-41-0P, (.+-.)-.alpha.-Tocopherol RL: IMF (Industrial manufacture); SPN (Synthetic preparation); PREP (Preparation) (process for the prepn. of .alpha.-tocopherol)				
IT	71-43-2, Benzene, uses 78-59-1, Isophorone 96-22-0, Diethyl ketone 96-48-0, .gamma.-Butyrolactone 96-49-1, Ethylene carbonate 105-58-8, Diethyl carbonate 108-10-1, Isobutyl methyl ketone 108-32-7, Propylene carbonate 108-88-3, Toluene, uses 110-54-3, Hexane, uses 111-65-9, Octane, uses 120-92-3, Cyclopentanone 142-82-5, Heptane, uses 616-38-6, Dimethyl carbonate 1330-20-7, Xylene, uses RL: NUU (Other use, unclassified); USES (Uses) (process for the prepn. of .alpha.-tocopherol)				
IT	144-62-7, Oxalic acid, reactions 700-13-0, Trimethylhydroquinone 7541-49-3 10026-13-8, Phosphorus pentachloride 60046-87-9 RL: RCT (Reactant); RACT (Reactant or reagent) (process for the prepn. of .alpha.-tocopherol)				

RE.CNT 3 THERE ARE 3 CITED REFERENCES AVAILABLE FOR THIS RECORD

RE

(1) Hoffmann La Roche; EP 0784042 A 1997 HCAPLUS

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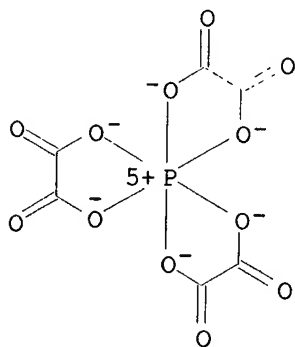
(3) Von Werder, F; US 2230659 A 1941 HCAPLUS

IT 321201-34-7P, Hydrogen tris(oxalato)phosphate(1-)

RL: CAT (Catalyst use); IMF (Industrial manufacture); SPN (Synthetic preparation); PREP (Preparation); USES (Uses)
(process for the prepn. of .alpha.-tocopherol)

RN 321201-34-7 HCAPLUS

CN Phosphate(1-), tris[ethanedioato(2-)-.kappa.O1,.kappa.O2]-, hydrogen, (OC-6-11)- (9CI) (CA INDEX NAME)



● H⁺

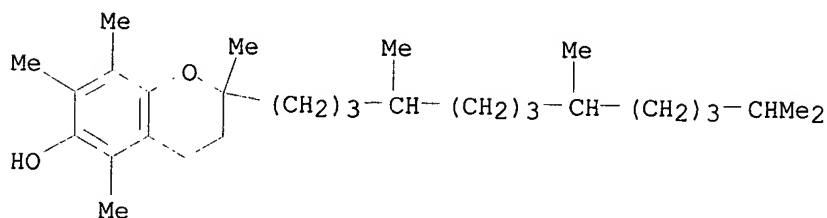
IT 10191-41-0P, (.+-.)-.alpha.-Tocopherol

RL: IMF (Industrial manufacture); SPN (Synthetic preparation); PREP (Preparation)

(process for the prepn. of .alpha.-tocopherol)

RN 10191-41-0 HCAPLUS

CN 2H-1-Benzopyran-6-ol, 3,4-dihydro-2,5,7,8-tetramethyl-2-(4,8,12-trimethyltridecyl)- (9CI) (CA INDEX NAME)



*Remaining prep's of
α tocopherol
starting
with
hydroquinone
+
phytol
and any
catalyst*

=> d que

L12

22 SEA FILE=REGISTRY ABB=ON (10026-13-8/BI OR 10191-41-0/BI OR 105-58-8/BI OR 108-10-1/BI OR 108-32-7/BI OR 108-88-3/BI OR 110-54-3/BI OR 111-65-9/BI OR 120-92-3/BI OR 1330-20-7/BI OR 142-82-5/BI OR 144-62-7/BI OR 321201-34-7/BI OR 60046-87-9/BI OR 616-38-6/BI OR 700-13-0/BI OR 71-43-2/BI OR 7541-49-3/BI OR 78-59-1/BI OR 96-22-0/BI OR 96-48-0/BI OR 96-49-1/BI)

L13

1 SEA FILE=REGISTRY ABB=ON 10191-41-0

L14

1032 SEA FILE=HCAPLUS ABB=ON L13

L15 104 SEA FILE=HCAPLUS ABB=ON L14(L) (PREP OR IMF OR SPN)/RL
 L16 2 SEA FILE=REGISTRY ABB=ON L12 AND 1/P
 L17 1 SEA FILE=REGISTRY ABB=ON L16 AND PHOSPHATE?
 L18 2 SEA FILE=HCAPLUS ABB=ON L17
 L19 1 SEA FILE=HCAPLUS ABB=ON L15 AND L18
 L20 59 SEA FILE=HCAPLUS ABB=ON L15 AND ?PHYTOL?
 L21 1 SEA FILE=REGISTRY ABB=ON L12 AND HYDROQUINONE
 L22 687 SEA FILE=HCAPLUS ABB=ON L21
 L23 48 SEA FILE=HCAPLUS ABB=ON L20 AND L22
 L24 24 SEA FILE=HCAPLUS ABB=ON L23 AND CAT/RL
 L25 23 SEA FILE=HCAPLUS ABB=ON L24 NOT L19

=> d 125 1-23 all

L25 ANSWER 1 OF 23 HCAPLUS COPYRIGHT 2003 ACS

AN 2002:332181 HCAPLUS

DN 136:340853

TI Method for preparing dl-alpha-tocopherol with high yield

IN Kim, Jeong-Soo; Cheong, Heui-Young; Lee, Sijoon

PA SK Corporation, S. Korea

SO PCT Int. Appl., 17 pp.

CODEN: PIXXD2

DT Patent

LA English

IC ICM C07D311-74

CC 30-20 (Terpenes and Terpenoids)

Section cross-reference(s): 67

FAN.CNT 1

	PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
PI	WO 2002034738	A1	20020502	WO 2000-KR1201	20001023
	W: CN, JP				
	RW: AT, BE, CH, CY, DE, DK, ES, FI, FR, GB, GR, IE, IT, LU, MC, NL, PT, SE				

	US 6384245	B1	20020507	US 2000-697615	20001026
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PRAI WO 2000-KR1201 A 20001023

OS CASREACT 136:340853

AB The invention discloses a method for prepg. DL-.alpha.-tocopherol (I) with a high yield through the condensation of **isophytol** or **phytol** derivs. with trimethylhydroquinone (TMHQ) in the presence of a Zn-Al heterogeneous catalyst system. At 80 to 120 .degree.C, the condensation is carried out for 2 to 7 h in the presence of a Zn(II) ion-coated alumina-silica catalyst in an n-heptane solvent. The synthetic Zn(II) ion-coated silica-alumina synthetic catalyst system can remarkably reduce side-reactions upon the condensation of **isophytol** or **phytol** derivs. and TMHQ, thus producing I with a high purity at a high yield. In addn., the catalyst system is greatly convenient to handle and therefore apply for continuous reactions for the prepn. of I. With these advantages, the catalyst system can be effectively used in prepg. highly pure I at a high yield on a com. scale.

ST tocopherol prepn zinc aluminum heterogeneous catalyst; **isophytol**
phytol deriv trimethylhydroquinone condensation tocopherol prepn

IT Tocopherols

RL: SPN (Synthetic preparation); PREP (Preparation)

(method for prepg. tocopherol via condensation of **isophytol** or **phytol** derivs. with trimethylhydroquinone in the presence of a Zn-Al heterogeneous catalyst system)

IT Catalysts

Condensation reaction catalysts

(zinc-aluminum; in prepg. tocopherol via condensation of **isophytol** or **phytol** derivs. with trimethylhydroquinone in the presence of a Zn-Al heterogeneous catalyst system)

IT 10191-41-0P, DL-.alpha.-Tocopherol

RL: IMF (Industrial manufacture); SPN (Synthetic preparation); PREP (Preparation)

(method for prepg. tocopherol via condensation of **isophytol** or **phytol** derivs. with trimethylhydroquinone in the presence of a Zn-Al heterogeneous catalyst system)

IT 150-86-7, **Phytol** 505-32-8, **Isophytol** 700-13-0, Trimethylhydroquinone 58425-36-8

RL: RCT (Reactant); RACT (Reactant or reagent)

(method for prepg. tocopherol via condensation of **isophytol** or **phytol** derivs. with trimethylhydroquinone in the presence of a Zn-Al heterogeneous catalyst system)

IT 7646-85-7, Zinc chloride, uses

RL: CAT (Catalyst use); USES (Uses)

(silica-alumina bound; method for prepg. tocopherol via condensation of **isophytol** or **phytol** derivs. with trimethylhydroquinone in the presence of a Zn-Al heterogeneous catalyst system)

IT 159995-97-8, Aluminum silicon oxide

RL: CAT (Catalyst use); USES (Uses)

(zinc chloride coated; method for prepg. tocopherol via condensation of **isophytol** or **phytol** derivs. with trimethylhydroquinone in the presence of a Zn-Al heterogeneous catalyst system)

IT 10236-16-5, **Phytol** acetate

RL: RCT (Reactant); RACT (Reactant or reagent)

(zinc chloride coated; method for prepg. tocopherol via condensation of **isophytol** or **phytol** derivs. with trimethylhydroquinone in the presence of a Zn-Al heterogeneous catalyst system)

RE.CNT 5 THERE ARE 5 CITED REFERENCES AVAILABLE FOR THIS RECORD

RE

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(2) Kuraray Co; US 4252726 A 1981 HCAPLUS

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L25 ANSWER 2 OF 23 HCAPLUS COPYRIGHT 2003 ACS

AN 2002:239437 HCAPLUS

DN 137:109397

TI Synthesis of (all-rac)-.alpha.-tocopherol using fluorinated NH-acidic catalysts

AU Bonrath, Werner; Haas, Alois; Hoppmann, Eike; Netscher, Thomas; Pauling, Horst; Schager, Frank; Wildermann, Angela

CS Research and Development, Roche Vitamins Ltd, Basel, 4070, Switz.

SO Advanced Synthesis & Catalysis (2002), 344(1), 37-39

CODEN: ASCAF7; ISSN: 1615-4150

PB Wiley-VCH Verlag GmbH

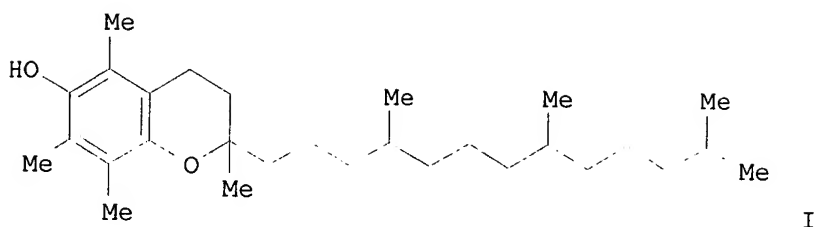
DT Journal

LA English

CC 30-30 (Terpenes and Terpenoids)

OS CASREACT 137:109397

GI



- AB The synthesis of (all-rac)-.alpha.-tocopherol (I) starting from trimethylhydroquinone and **isophytol** using fluorinated NH-acidic catalysts is described. The scope and limitations of this type of catalyst are discussed. The advantages of this new procedure are high yield and selectivity, no waste problem and mild reaction conditions. The best results in the synthesis of I (94% yield) using NH-acidic compds. are obtained in polar solvents. The used catalyst could be recovered.
- ST tocopherol prepn trimethylhydroquinone **isophytol** Friedel Crafts alkylation biphasic catalyst; ring closure catalyst biphasic tocopherol prepn trimethylhydroquinone **isophytol**; fluorinated NH acid catalyst tocopherol prepn
- IT Cyclization catalysts
Friedel-Crafts reaction catalysts
(synthesis of (all-rac)-.alpha.-tocopherol using fluorinated NH-acidic catalysts)
- IT Cyclization
Friedel-Crafts reaction
(synthesis of (all-rac)-.alpha.-tocopherol using fluorinated NH-acidic catalysts via)
- IT 1535-93-9 39847-37-5 39847-38-6 39847-39-7 39847-40-0 39847-41-1
82113-65-3 84246-29-7 152894-10-5 152894-12-7 200728-68-3
400608-35-7 400608-36-8
RL: **CAT (Catalyst use)**; **USES (Uses)**
(synthesis of (all-rac)-.alpha.-tocopherol using fluorinated NH-acidic catalysts)
- IT 96-22-0, Diethyl ketone 96-48-0, .gamma.-Butyrolactone 96-49-1,
Ethylene carbonate 108-32-7, Propylene carbonate 108-88-3, Toluene,
uses 142-82-5, Heptane, uses
RL: **NUU (Other use, unclassified)**; **USES (Uses)**
(synthesis of (all-rac)-.alpha.-tocopherol using fluorinated NH-acidic catalysts)
- IT **700-13-0** 60046-87-9 - *hydroquinone*
RL: **RCT (Reactant)**; **RAC (Reactant or reagent)**
(synthesis of (all-rac)-.alpha.-tocopherol using fluorinated NH-acidic catalysts)
- IT **10191-41-0P** *alpha tocopherol*
RL: **RCT (Reactant)**; **SPN (Synthetic preparation)**; **PREP (Preparation)**; **RAC (Reactant or reagent)**
(synthesis of (all-rac)-.alpha.-tocopherol using fluorinated NH-acidic catalysts)
- IT 52225-20-4P
RL: **SPN (Synthetic preparation)**; **PREP (Preparation)**
(synthesis of (all-rac)-.alpha.-tocopherol using fluorinated NH-acidic catalysts)
- RE.CNT 25 THERE ARE 25 CITED REFERENCES AVAILABLE FOR THIS RECORD
RE
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- (2) Aquino, F; Appl Catal A: General 2001, V220, P51
(3) Baak, M; WO 9821197 1998 HCAPLUS
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(15) March, J; Advanced Organic Chemistry, 4th Ed 1992, P1011
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(24) Schulz, B; DE 3203487 A1 1983 HCAPLUS
(25) Wang, S; J Supercrit Fluids 2000, V17, P135 HCAPLUS
- L25 ANSWER 3 OF 23 HCAPLUS COPYRIGHT 2003 ACS
AN 2002:138923 HCAPLUS
DN 136:183967
TI Process for manufacturing all-rac.-.alpha.-tocopherol in the presence of a bis(perfluoroalkanesulfonyl or pentafluorobenzenesulfonyl)imide catalyst
IN Bonrath, Werner; Haas, Alois; Hoppmann, Eike; Pauling, Horst
PA Roche Vitamins A.-G., Switz.
SO Eur. Pat. Appl., 10 pp.
CODEN: EPXXDW
DT Patent
LA English
IC ICM C07D311-72
CC 30-20 (Terpenes and Terpenoids)
Section cross-reference(s): 67
FAN.CNT 1
- | | PATENT NO. | KIND | DATE | APPLICATION NO. | DATE |
|------|--|------|----------|-----------------|----------|
| | ----- | ---- | ----- | ----- | ----- |
| PI | EP 1180517 | A1 | 20020220 | EP 2001-119322 | 20010810 |
| | R: AT, BE, CH, DE, DK, ES, FR, GB, GR, IT, LI, LU, NL, SE, MC, PT, IE, SI, LT, LV, FI, RO | | | | |
| | BR 2001003412 | A | 20020326 | BR 2001-3412 | 20010816 |
| | JP 2002128775 | A2 | 20020509 | JP 2001-246843 | 20010816 |
| | US 2002072619 | A1 | 20020613 | US 2001-931663 | 20010816 |
| | US 6482961 | B2 | 20021119 | | |
| | CN 1339437 | A | 20020313 | CN 2001-125595 | 20010817 |
| PRAI | EP 2000-117761 | A | 20000818 | | |
| OS | CASREACT 136:183967; MARPAT 136:183967 | | | | |
| AB | A process, for the manuf. of all-racemic-.alpha.-tocopherol (I) via the acid-catalyzed condensation of trimethylhydroquinone with phytol or isophytol , is characterized by carrying out the condensation in the presence of a bis(perfluorohydrocarbyl)imide or a metal bis(perfluorohydrocarbyl)imide, [(R1SO2)3N]xR2 [R1 = perfluoroalkyl, CnF2n+1, pentafluorophenyl; R2 = H, B, Mg, Al, Si, Sc, Ti, V, VO, Mn, Fe, Co, Ni, Cu, Zn, Y, Zr, Rh, Pd, Ag, Sn, La, Ce, Pr, Nd, Eu, Dy, Yb, Hf, Pt, | | | | |

Au; n = 1 - 10; x = valency of proton (1) or metal cation (1-4)], as a catalyst in an org. solvent. Thus, trimethylhydroquinone was suspended in PhMe and catalytic bis(pentafluoroethanesulfonyl)amine was added, after heating to 50-150.degree., **isophytol** was added over 20 mins., after 30 mins. more, the solvent is removed, giving 89.6% crude I.

- ST tocopherol alpha manufg process; trisperfluoroalkanesulfonyl catalyst manufg process alpha tocopherol; trimethylhydroquinone condensation **isophytol phytol** bissulfonylimide catalyst; imide bisperfluoroalkanesulfonyl catalyst manufg process alpha tocopherol
- IT Hydrocarbons, uses
 RL: NUU (Other use, unclassified); USES (Uses)
 (aliph. or arom., solvent; process for manufg. all-rac.-.alpha.-tocopherol by acid-catalyzed condensation of trimethylhydroquinone with **phytol** or **isophytol** in the presence of a bis(perfluoroalkanesulfonyl)imide catalyst)
- IT Ketones, uses
 RL: NUU (Other use, unclassified); USES (Uses)
 (aliph. or cyclic, solvent; process for manufg. all-rac.-.alpha.-tocopherol by acid-catalyzed condensation of trimethylhydroquinone with **phytol** or **isophytol** in the presence of a bis(perfluoroalkanesulfonyl)imide catalyst)
- IT Bronsted acids
 RL: CAT (Catalyst use); USES (Uses)
 (co-catalyst; process for manufg. all-rac.-.alpha.-tocopherol by acid-catalyzed condensation of trimethylhydroquinone with **phytol** or **isophytol** in the presence of a bis(perfluoroalkanesulfonyl)imide catalyst)
- IT Carboxylic acids, uses
 RL: NUU (Other use, unclassified); USES (Uses)
 (esters, aliph. or cyclic, solvent; process for manufg. all-rac.-.alpha.-tocopherol by acid-catalyzed condensation of trimethylhydroquinone with **phytol** or **isophytol** in the presence of a bis(perfluoroalkanesulfonyl)imide catalyst)
- IT Condensation reaction
 Condensation reaction catalysts
 (process for manufg. all-rac.-.alpha.-tocopherol by acid-catalyzed condensation of trimethylhydroquinone with **phytol** or **isophytol** in the presence of a bis(perfluoroalkanesulfonyl or pentafluorobenzenesulfonyl)imide catalyst)
- IT Metals, uses
 RL: CAT (Catalyst use); USES (Uses)
 (trisulfonylimide catalysts; process for manufg. all-rac.-.alpha.-tocopherol by acid-catalyzed condensation of trimethylhydroquinone with **phytol** or **isophytol** in the presence of a bis(perfluoroalkanesulfonyl)imide catalyst)
- IT 1535-93-9 39847-37-5 39847-38-6 39847-39-7 39847-40-0 39847-41-1
 84246-29-7 84331-53-3 152894-10-5 152894-12-7 192888-07-6
 200728-68-3 213342-85-9 296280-11-0 400608-35-7 400608-36-8
 400608-37-9 400629-85-8 400629-88-1 400629-90-5 400630-94-6
 400630-96-8
 RL: CAT (Catalyst use); USES (Uses)
 (process for manufg. all-rac.-.alpha.-tocopherol by acid-catalyzed condensation of trimethylhydroquinone with **phytol** or **isophytol** in the presence of a bis(perfluoroalkanesulfonyl or pentafluorobenzenesulfonyl)imide catalyst)
- IT 10191-41-0P
 RL: IMF (Industrial manufacture); SPN (Synthetic preparation); PREP (Preparation)
 (process for manufg. all-rac.-.alpha.-tocopherol by acid-catalyzed condensation of trimethylhydroquinone with **phytol** or

isophytol in the presence of a bis(perfluoroalkanesulfonyl or pentafluorobenzenesulfonyl)imide catalyst)

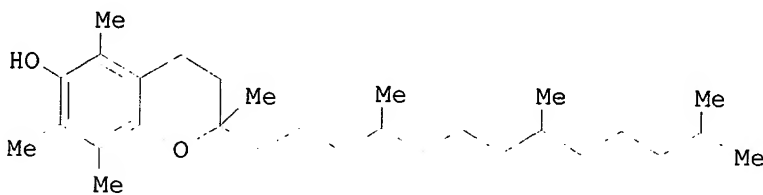
IT 150-86-7, **Phytol** 505-32-8, **Isophytol**
700-13-0, Trimethylhydroquinone
RL: RCT (Reactant); RACT (Reactant or reagent)
(process for manufg. all-rac.-.alpha.-tocopherol by acid-catalyzed condensation of trimethylhydroquinone with **phytol** or **isophytol** in the presence of a bis(perfluoroalkanesulfonyl or pentafluorobenzenesulfonyl)imide catalyst)

IT 71-43-2, Benzene, uses 78-59-1, Isophorone 96-22-0, Diethyl ketone 96-48-0, .gamma.-Butyrolactone 96-49-1, Ethylene carbonate 108-10-1, Isobutyl methyl ketone 108-21-4, Isopropyl acetate 108-32-7, Propylene carbonate 108-88-3, Toluene, uses 110-54-3, Hexane, uses 111-65-9, Octane, uses 120-92-3, Cyclopentanone 141-78-6, Ethyl acetate, uses 142-82-5, Heptane, uses 1330-20-7, Xylene, uses
RL: NUU (Other use, unclassified); USES (Uses)
(solvent; process for manufg. all-rac.-.alpha.-tocopherol by acid-catalyzed condensation of trimethylhydroquinone with **phytol** or **isophytol** in the presence of a bis(perfluoroalkanesulfonyl or pentafluorobenzenesulfonyl)imide catalyst)

RE.CNT 3 THERE ARE 3 CITED REFERENCES AVAILABLE FOR THIS RECORD
RE
(1) Hoffman La Roche; WO 9821197 A 1998 HCAPLUS
(2) Hoffman La Roche; EP 1000940 A 2000 HCAPLUS
(3) Kazuaki, I; SYNLETT 1996, P1045

L25 ANSWER 4 OF 23 HCAPLUS COPYRIGHT 2003 ACS
AN 2001:691767 HCAPLUS
DN 135:257368
TI Process for manufacturing all-rac.-.alpha.-tocopherol in the presence of a tris(perfluoroalkanesulfonyl or pentafluorobenzenesulfonyl)methane catalyst
IN Bonrath, Werner; Haas, Alois; Hoppmann, Eike; Netscher, Thomas; Pauling, Horst
PA F. Hoffmann-La Roche A.-G., Switz.
SO Eur. Pat. Appl., 9 pp.
CODEN: EPXXDW
DT Patent
LA English
IC ICM C07D311-72
CC 30-20 (Terpenes and Terpenoids)
Section cross-reference(s): 29, 67
FAN.CNT 1

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
-----	---	-----	-----	-----
PI EP 1134218	A1	20010919	EP 2001-105979	20010310
R: AT, BE, CH, DE, DK, ES, FR, GB, GR, IT, LI, LU, NL, SE, MC, PT, IE, SI, LT, LV, FI, RO				
US 2001027255	A1	20011004	US 2001-802272	20010308
US 6369242	B2	20020409		
CA 2340518	AA	20010917	CA 2001-2340518	20010313
JP 2001294584	A2	20011023	JP 2001-71442	20010314
BR 2001001075	A	20011106	BR 2001-1075	20010315
CN 1314354	A	20010926	CN 2001-111398	20010316
PRAI EP 2000-105672	A	20000317		
OS CASREACT 135:257368; MARPAT 135:257368				
GI				



I

- AB A process, for the manuf. of all-racemic- α -tocopherol (I) via the acid-catalyzed condensation of trimethylhydroquinone with **phytol** or **isophytol**, is characterized by carrying out the condensation in the presence of a tris(perfluoroalkanesulfonyl or pentafluorobenzenesulfonyl)methane or a metal tris(perfluoroalkanesulfonyl or pentafluorobenzenesulfonyl)methide, $[(R_1SO_2)_3C] \times R_2$ [R_1 = perfluoroalkyl, C_nF_{2n+1} , pentafluorophenyl; R_2 = H, B, Mg, Al, Si, Sc, Ti, V, VO, Mn, Fe, Co, Ni, Cu, Zn, Y, Zr, Rh, Pd, Ag, Sn, La, Ce, Pr, Nd, Eu, Dy, Yb, Hf, Pt, Au; $n = 1 - 10$; x = valency of proton (1) or metal cation (1 - 4)], as a catalyst in an org. solvent. Thus, trimethylhydroquinone was suspended in PhMe and catalytic tris(trifluoromethanesulfonyl)methane was added, after heating to 100.degree., **isophytol** was added over 60 mins., after 30 mins. more, the solvent is removed, giving 91.7% crude I.
- ST tocopherol α manufg process; trispentafluorobenzenesulfonylmethane catalyst manufg process α tocopherol; trisperfluoroalkanesulfonylmethane catalyst manufg process α tocopherol; trimethylhydroquinone condensation **isophytol** **phytol** trisulfonylmethane catalyst; tristrifluoromethanesulfonylmethane catalyst manufg process α tocopherol; methide trisperfluoroalkanesulfonyl catalyst manufg process α tocopherol
- IT Hydrocarbons, uses
RL: NUU (Other use, unclassified); USES (Uses)
(aliph. or arom., solvent; prepn. of all-rac.- α -tocopherol via condensation of trimethylhydroquinone with **isophytol** in the presence of a trisulfonylmethane catalyst)
- IT Ketones, uses
RL: NUU (Other use, unclassified); USES (Uses)
(aliph. or cyclic, solvent; prepn. of all-rac.- α -tocopherol via condensation of trimethylhydroquinone with **isophytol** in the presence of a trisulfonylmethane catalyst)
- IT Bronsted acids
RL: CAT (Catalyst use); USES (Uses)
(co-catalyst; prepn. of all-rac.- α -tocopherol via condensation of trimethylhydroquinone with **isophytol** in the presence of a trisulfonylmethane catalyst)
- IT Carboxylic acids, uses
RL: NUU (Other use, unclassified); USES (Uses)
(esters, aliph. or cyclic, solvent; prepn. of all-rac.- α -tocopherol via condensation of trimethylhydroquinone with **isophytol** in the presence of a trisulfonylmethane catalyst)
- IT Solvents
(org.; prepn. of all-rac.- α -tocopherol via condensation of trimethylhydroquinone with **isophytol** in the presence of a trisulfonylmethane catalyst)
- IT Condensation reaction
Condensation reaction catalysts
(prepn. of all-rac.- α -tocopherol via condensation of trimethylhydroquinone with **isophytol** in the presence of a

trisulfonylmethane catalyst)

IT Metals, uses
RL: CAT (Catalyst use); USES (Uses)
(trisulfonylmethide catalysts; prepn. of all-rac.-alpha-tocopherol via condensation of trimethylhydroquinone with **isophytol** in the presence of a trisulfonylmethane catalyst)

IT 7664-38-2, Phosphoric acid, uses 7664-93-9, Sulfuric acid, uses
RL: CAT (Catalyst use); USES (Uses)
(co-catalyst; prepn. of all-rac.-alpha-tocopherol via condensation of trimethylhydroquinone with **isophytol** in the presence of a trisulfonylmethane catalyst)

IT 104-15-4, p-Toluenesulfonic acid, uses 60805-12-1,
Tris(trifluoromethanesulfonyl)methane 114395-71-0 261728-13-6
360574-09-0 360574-11-4 360574-12-5D, metal salts 361343-17-1
361343-18-2
RL: CAT (Catalyst use); USES (Uses)
(prepn. of all-rac.-alpha-tocopherol via condensation of trimethylhydroquinone with **isophytol** in the presence of a trisulfonylmethane catalyst)

IT 10191-41-0P, (.+-.)-.alpha.-Tocopherol
RL: IMF (Industrial manufacture); SPN (Synthetic preparation); PREP (Preparation)
(prepn. of all-rac.-alpha-tocopherol via condensation of trimethylhydroquinone with **isophytol** in the presence of a trisulfonylmethane catalyst)

IT 150-86-7, **Phytol** 505-32-8, **Isophytol**
700-13-0, 2,3,5-Trimethyl-1,4-hydroquinone
RL: RCT (Reactant); RACT (Reactant or reagent)
(prepn. of all-rac.-alpha-tocopherol via condensation of trimethylhydroquinone with **isophytol** in the presence of a trisulfonylmethane catalyst)

IT 71-43-2, Benzene, uses 78-59-1, Isophorone 96-22-0, Diethyl ketone 96-48-0, .gamma.-Butyrolactone 96-49-1, Ethylene carbonate 108-10-1, Isobutyl methyl ketone 108-21-4, Isopropyl acetate 108-32-7, Propylene carbonate 108-88-3, Toluene, uses 110-54-3, Hexane, uses 111-65-9, Octane, uses 120-92-3, Cyclopentanone 141-78-6, Ethyl acetate, uses 142-82-5, Heptane, uses 1330-20-7, Xylene, uses
RL: NUU (Other use, unclassified); USES (Uses)
(solvent; prepn. of all-rac.-alpha-tocopherol via condensation of trimethylhydroquinone with **isophytol** in the presence of a trisulfonylmethane catalyst)

RE.CNT 5 THERE ARE 5 CITED REFERENCES AVAILABLE FOR THIS RECORD
RE

(1) Eisai Co; EP 0658552 A 1995 HCAPLUS
(2) Hoffmann-La Roche, F; WO 9821197 A 1998 HCAPLUS
(3) Hoffmann-La Roche, F; EP 0949255 A 1999 HCAPLUS
(4) Hoffmann-La Roche, F; EP 1000940 A 2000 HCAPLUS
(5) Minnesota Mining And Manufacturing Company; US 5554664 A 1996 HCAPLUS

L25 ANSWER 5 OF 23 HCAPLUS COPYRIGHT 2003 ACS
AN 2001:453047 HCAPLUS
DN 135:61459
TI Method for preparing dl-alpha-tocopherol with a high yield and high purity
IN Lee, Sijoon; Kim, Jeong-soo; Yoon, Young-seek; Kim, Myung-jun; Choi, Jun-tae; Kwak, Byong-sung
PA SK Corp., S. Korea
SO PCT Int. Appl., 28 pp.
CODEN: PIXXD2
DT Patent
LA English

IC ICM C07D311-74

CC 30-20 (Terpenes and Terpenoids)

Section cross-reference(s): 67

FAN.CNT 1

	PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
	-----	----	-----	-----	-----
PI	WO 2001044224	A1	20010621	WO 2000-KR1314	20001116
	W: CN, JP				
	RW: AT, BE, CH, CY, DE, DK, ES, FI, FR, GB, GR, IE, IT, LU, MC, NL, PT, SE, TR				
	US 6441200	B1	20020827	US 2000-711824	20001113
	EP 1237885	A1	20020911	EP 2000-981848	20001116
	R: AT, BE, CH, DE, DK, ES, FR, GB, GR, IT, LI, LU, NL, SE, MC, PT, IE, SI, LT, LV, FI, RO, MK, CY, AL, TR				
PRAI	KR 1999-57483	A	19991214		
	WO 2000-KR1314	W	20001116		
OS	CASREACT 135:61459				
AB	Disclosed is a method for prepg. DL-.alpha.-tocopherol through the condensation of isophytol or phytol derivs. and trimethylhydroquinone(TMHQ) using a catalyst system comprising a divalent metal halogen compd., silica gel and/or silica-alumina, and a Bronsted acid. Isophytol or phytol derivs. are slowly added to trimethylhydroquinone for the condensation thereof at 80 to 135 .degree.C over 30 to 60 min in the presence of the metal halogen and the silica gel and/or silica-alumina. In the presence of the Bronsted acid, the intermediates are converted into the product. The silica gel and/or silica-alumina is washed with a polar solvent for recovery. The catalyst system can remarkably reduce side-reactions upon the condensation of isophytol or phytol derivs. and TMHQ, thus producing DL-.alpha.-tocopherol with a high purity at a high yield. Also, the catalyst system can be regenerated in succession because of its being able to avoid the decrease of catalytic activity attributed to the adsorption of org. materials; thus reducing the prodn. cost of DL-.alpha.-tocopherol and the quantity of industrial wastes generated. With these advantages, the catalyst system can be effectively used in prepg. highly pure DL-.alpha.-tocopherol at a high yield on a com. scale.				
ST	DL alpha tocopherol synthesis; cyclocondensation isophytol trimethylhydroquinone metal halide catalyzed				
IT	Cyclocondensation reaction catalysts (divalent metal halide catalyst in cyclocondensation of isophytol and trimethylhydroquinone in prepn. of DL-alpha-tocopherol)				
IT	Chlorides, uses Fluorides, uses Iodides, uses RL: CAT (Catalyst use) ; USES (Uses) (divalent; prepn. of DL-.alpha.-tocopherol with high yield and high purity)				
IT	Alcohols, preparation Diterpenes RL: SPN (Synthetic preparation) ; PREP (Preparation) (hydroxy diterpenes; prepn. of DL-.alpha.-tocopherol with high yield and high purity)				
IT	Cyclocondensation reaction (prepn. of DL-.alpha.-tocopherol with high yield and high purity)				
IT	Silica gel, uses RL: CAT (Catalyst use) ; USES (Uses) (prepn. of DL-.alpha.-tocopherol with high yield and high purity)				
IT	1344-28-1, Alumina, uses 7646-85-7, Zinc dichloride, uses 15438-31-0D, Iron dication, halide, uses 22541-90-8D, Tin dication, halide, uses				

23713-49-7D, Zinc dication, halide, uses
RL: CAT (Catalyst use); USES (Uses)
(prepn. of DL-.alpha.-tocopherol with high yield and high purity)
IT 10191-41-0P, dl-.alpha.-Tocopherol
RL: IMF (Industrial manufacture); SPN (Synthetic preparation); PREP (Preparation)
(prepn. of DL-.alpha.-tocopherol with high yield and high purity)
IT 64-17-5, Ethanol, uses 67-56-1, Methanol, uses 67-63-0, Isopropyl alcohol, uses 67-64-1, Acetone, uses 71-23-8, Propanol, uses 71-36-3, Butanol, uses 71-41-0, Pentanol, uses 71-43-2, Benzene, uses 75-09-2, Dichloromethane, uses 78-93-3, Methyl ethyl ketone, uses 79-20-9, Methyl acetate 96-22-0, Diethyl ketone 105-53-3, Diethyl malonate 108-88-3, Toluene, uses 109-60-4, Propyl acetate 110-54-3, n-Hexane, uses 111-27-3, Hexanol, uses 111-65-9, n-Octane, uses 111-70-6, 1-Heptanol 111-87-5, Octanol, uses 123-86-4, Butyl acetate 124-18-5, n-Decane 141-78-6, Ethyl acetate, uses 142-82-5, n-Heptane, uses 563-80-4, Methyl isopropyl ketone 628-63-7, Pentyl acetate 1300-21-6, Dichloroethane 1330-20-7, Xylene, uses 26635-64-3, Isooctane 31394-54-4, Isoheptane 34464-38-5, Isodecane 43133-95-5, Methylpentane
RL: NUU (Other use, unclassified); USES (Uses)
(prepn. of DL-.alpha.-tocopherol with high yield and high purity)
IT 150-86-7D, Phytol, derivs. 505-32-8, Isophytol
700-13-0, Trimethylhydroquinone
RL: RCT (Reactant); RACT (Reactant or reagent)
(prepn. of DL-.alpha.-tocopherol with high yield and high purity)
IT 104-15-4P, p-Toluenesulfonic acid, preparation 7647-01-0P, Hydrochloric acid, preparation 7664-38-2P, Phosphoric acid, preparation 7664-93-9P, Sulfuric acid, preparation 7697-37-2P, Nitric acid, preparation
RL: SPN (Synthetic preparation); PREP (Preparation)
(prepn. of DL-.alpha.-tocopherol with high yield and high purity)
RE.CNT 4 THERE ARE 4 CITED REFERENCES AVAILABLE FOR THIS RECORD
RE
(1) Basf Ag; US 5468883 A 1995 HCAPLUS
(2) Kuraray Co; US 4252726 A 1981 HCAPLUS
(3) Mitsui Toatsu Chem Inc; JP 59190987 A 1984 HCAPLUS
(4) Nisshin Flour Milling Co Ltd; JP 53-44570 A 1978 HCAPLUS

L25 ANSWER 6 OF 23 HCAPLUS COPYRIGHT 2003 ACS
AN 2000:360971 HCAPLUS
DN 133:150747
TI Synthesis of d,l-.alpha.-tocopherol using 'microencapsulated' catalysts
AU Schager, F.; Bonrath, W.
CS Vitamin Research and Technology Development, F. Hoffmann-La Roche Ltd., Basel, CH-4070, Switz.
SO Applied Catalysis, A: General (2000), 202(1), 117-120
CODEN: ACAGE4; ISSN: 0926-860X
PB Elsevier Science B.V.
DT Journal
LA English
CC 30-30 (Terpenes and Terpenoids)
Section cross-reference(s): 67
OS CASREACT 133:150747
AB The synthesis of d,l-.alpha.-tocopherol starting from trimethylhydroquinone and isophytol using 'microencapsulated' catalysts, e.g. MC-(CF3SO2)2NH, is described. Scope and limitations of this type of catalysts are discussed. Advantages of this new procedure are high yield and selectivity, no waste problem and mild reaction conditions. Best results in the synthesis of d,l-.alpha.-tocopherol using MC-catalysts are obtained in polar solvents. The used catalyst could not

be recovered.

ST tocopherol alpha prepn microencapsulated catalyst

IT Cyclocondensation reaction catalysts
(synthesis of d,l-.alpha.-tocopherol using 'microencapsulated' catalysts)

IT 82113-65-3 144026-79-9, Scandium triflate

RL: CAT (Catalyst use); USES (Uses)

(microencapsulated; synthesis of d,l-.alpha.-tocopherol using 'microencapsulated' catalysts)

IT 505-32-8, Isophytol 700-13-0, Trimethylhydroquinone

RL: RCT (Reactant); RACT (Reactant or reagent)

(synthesis of d,l-.alpha.-tocopherol using 'microencapsulated' catalysts)

IT 10191-41-0P, (.+-.)-.alpha.-Tocopherol

RL: SPN (Synthetic preparation); PREP (Preparation)

(synthesis of d,l-.alpha.-tocopherol using 'microencapsulated' catalysts)

RE.CNT 15 THERE ARE 15 CITED REFERENCES AVAILABLE FOR THIS RECORD

RE

(1) Baak, M; WO 9821197 1996 HCAPLUS

(2) Baldenius, K; Ullmann's Encyclopedia of Industrial Chemistry, 5th Edition 1996, VA27, P478

(3) Finnan, J; EP 100471 1982 HCAPLUS

(4) Greenbaum, S; US 3708505 1971 HCAPLUS

(5) Isler, O; Vitamine II 1988, P126

(6) Karrer, P; US 2411967 1946 HCAPLUS

(7) Kobayashi, S; J Am Chem Soc 1998, V120, P2985 HCAPLUS

(8) March, J; Advanced Organic Chemistry, 3rd Edition 1985, P902

(9) Matsui, M; EP 658552 A1 1993 HCAPLUS

(10) Matsui, M; Bull Chem Soc Jpn 1995, V68, P3569 HCAPLUS

(11) Nagayama, S; J Org Chem 1998, V63, P6094 HCAPLUS

(12) Nelan, D; US 3444213 1969 HCAPLUS

(13) Schager, F; J Catal 1999, V182, P284

(14) Schulz, B; DE 3203487 1983 HCAPLUS

(15) Skoda, A; CS 254402 1989

L25 ANSWER 7 OF 23 HCAPLUS COPYRIGHT 2003 ACS

AN 2000:335071 HCAPLUS

DN 132:334646

TI Process for manufacturing d,l-.alpha.-tocopherol

IN Bonrath, Werner; Wang, Shaoning

PA F. Hoffmann-La Roche A.-G., Switz.

SO Eur. Pat. Appl., 8 pp.

CODEN: EPXXDW

DT Patent

LA English

IC ICM C07D311-72

CC 30-20 (Terpenes and Terpenoids)

FAN.CNT 1

	PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
PI	EP 1000940	A1	20000517	EP 1999-121898	19991105
	R: AT, BE, CH, DE, DK, ES, FR, GB, GR, IT, LI, LU, NL, SE, MC, PT, IE, SI, LT, LV, FI, RO				
	KR 2000035335	A	20000626	KR 1999-49441	19991109
	CN 1253950	A	20000524	CN 1999-123521	19991110
	JP 2000143656	A2	20000526	JP 1999-319283	19991110
	BR 9905319	A	20010417	BR 1999-5319	19991110
	US 2002010347	A1	20020124	US 2001-932519	20010817
	US 6423851	B2	20020723		

PRAI EP 1998-121457 A 19981111
US 1999-438711 B1 19991111
OS CASREACT 132:334646
AB The manuf. of d,l-.alpha.-tocopherol by the catalyzed condensation of trimethylhydroquinone with **isophytol** comprises carrying out the condensation in the presence of bis-(trifluoromethylsulfonyl)amine, or a metal salt thereof of formula $M[N(SO_2CF_3)_2]_n$ [M = metal; n = 1-4], in supercrit. carbon dioxide or nitrous oxide as the solvent. Thus, trimethylhydroquinone, **isophytol** and bis(trifluoromethanesulfonyl)amine in supercrit. carbon dioxide were mixed in a stainless steel autoclave and heated to 150.degree.C at 85 bar to give dl-.alpha.-tocopherol in 84.23% yield.
ST tocopherol prepn condensation trifluoromethylsulfonylamine catalyst
IT Condensation reaction
Condensation reaction catalysts
(prepn. of d,l-.alpha.-tocopherol via trifluoromethanesulfonylamine catalyzed condensation)
IT 64-17-5, Ethanol, uses 67-56-1, Methanol, uses 67-64-1, Acetone, uses 74-98-6, Propane, uses 96-22-0, Diethyl ketone 108-10-1, Isobutyl methyl ketone
RL: NUU (Other use, unclassified); USES (Uses)
(cosolvent; prepn. of d,l-.alpha.-tocopherol via trifluoromethanesulfonylamine catalyzed condensation)
IT 82113-65-3, Bis(trifluoromethylsulfonyl)amine 189114-61-2, Silver bis(trifluoromethanesulfonyl)amide
RL: CAT (Catalyst use); USES (Uses)
(prepn. of d,l-.alpha.-tocopherol via trifluoromethanesulfonylamine catalyzed condensation)
IT 10191-41-0P, dl-.alpha.-Tocopherol
RL: IMF (Industrial manufacture); SPN (Synthetic preparation); PREP (Preparation)
(prepn. of d,l-.alpha.-tocopherol via trifluoromethanesulfonylamine catalyzed condensation)
IT 505-32-8, **Isophytol** 700-13-0, Trimethylhydroquinone
RL: RCT (Reactant); RACT (Reactant or reagent)
(prepn. of d,l-.alpha.-tocopherol via trifluoromethanesulfonylamine catalyzed condensation)
IT 124-38-9, Carbon dioxide, uses 10024-97-2, Nitrous oxide, uses
RL: NUU (Other use, unclassified); USES (Uses)
(solvent; prepn. of d,l-.alpha.-tocopherol via trifluoromethanesulfonylamine catalyzed condensation)
RE.CNT 5 THERE ARE 5 CITED REFERENCES AVAILABLE FOR THIS RECORD
RE
(1) Anon; PATENT ABSTRACTS OF JAPAN 1985, V009(309)
(2) Eisai Co Ltd; EP 0658552 A 1995 HCAPLUS
(3) F Hoffmann-La Roche Ag; WO 9821197 A 1998 HCAPLUS
(4) Ishihara, K; SYNLETT 11, P1045 HCAPLUS
(5) Mitsui Petrochem Ind K K; JP 60149582 A 1985 HCAPLUS
L25 ANSWER 8 OF 23 HCAPLUS COPYRIGHT 2003 ACS
AN 2000:256793 HCAPLUS
DN 133:30610
TI The synthesis of D,L-.alpha.-tocopherol in supercritical media
AU Wang, Shaoning; Bonrath, Werner; Pauling, Horst; Kienzle, Frank
CS F. Hoffmann-La Roche Ltd., Basel, Switz.
SO Journal of Supercritical Fluids (2000), 17(2), 135-143
CODEN: JSFLEH; ISSN: 0896-8446
PB Elsevier Science B.V.
DT Journal
LA English

CC 26-8 (Biomolecules and Their Synthetic Analogs)
OS CASREACT 133:30610
AB The synthesis of D,L-.alpha.-tocopherol in supercrit. carbon dioxide or nitrous oxide by condensation of trimethylhydroquinone (TMHQ) with **isophytol** (IP) in the presence of various Bronsted or Lewis acids, esp. an imide or its metal salts, as catalysts is described. The product is obtained in high yield. The method represents an interesting alternative to existing processes. A quasi-reaction mechanism is being proposed together with kinetics, which are needed for the reactor anal. and design.
ST Bronsted Lewis acid cyclocondensation trimethylhydroquinone **isophytol**; tocopherol prepn Bronsted Lewis acid cyclocondensation; vitamin E prepn Bronsted Lewis cyclocondensation
IT Cyclocondensation reaction
Cyclocondensation reaction catalysts
(prepn. of D,L-.alpha.-tocopherol in supercrit. media)
IT Bronsted acids
Lewis acids
RL: **CAT (Catalyst use)**; **USES (Uses)**
(prepn. of D,L-.alpha.-tocopherol in supercrit. media)
IT 1343-93-7, 12-Tungstophosphoric acid (H3PW12O40) 9037-24-5, Amberlyst-15 82113-65-3, Bis(trifluoromethanesulfonyl)amine 90076-65-6, Lithium bis(trifluoromethylsulfonyl)amide 118473-68-0, Nafion NR50 189114-61-2, Silver bis(trifluoromethylsulfonyl)amide
RL: **CAT (Catalyst use)**; **USES (Uses)**
(prepn. of D,L-.alpha.-tocopherol in supercrit. media)
IT 124-38-9, Carbon dioxide, uses 10024-97-2, Nitrogen oxide (N2O), uses
RL: **NUU (Other use, unclassified)**; **USES (Uses)**
(prepn. of D,L-.alpha.-tocopherol in supercrit. media)
IT 505-32-8, **Isophytol** 700-13-0, Trimethylhydroquinone
RL: **RCT (Reactant)**; **RACT (Reactant or reagent)**
(prepn. of D,L-.alpha.-tocopherol in supercrit. media)
IT **10191-41-0P**, D,L-.alpha.-Tocopherol
RL: **SPN (Synthetic preparation)**; **PREP (Preparation)**
(prepn. of D,L-.alpha.-tocopherol in supercrit. media)
RE.CNT 13 THERE ARE 13 CITED REFERENCES AVAILABLE FOR THIS RECORD
RE
(1) Anon; Chemical Market Reporter 1997, P5
(2) Baldenius, K; Ullmann's Encyclopedia of Industrial Chemistry 1996, VA27, P484
(3) Bonrath, W; European Patent Application 1998
(4) Brunner, G; J Supercritical Fluids 1991, V4, P72 HCAPLUS
(5) Koga, Y; Fluid Phase Equilibria 1996, V125, P115 HCAPLUS
(6) Lowack, R; EP 0603695 A1 1994 HCAPLUS
(7) Mayer, H; Helv Chim Acta 1963, V46, P650 HCAPLUS
(8) Mayer, H; Helv Chim Acta 1967, V139, P1376
(9) Press, W; Numerical Recipes 1986
(10) Schudel, P; Helv Chim Acta 1963, V46, P2517 HCAPLUS
(11) Wang, S; Chem Eng Sci 1999, V54, P1639 HCAPLUS
(12) Wang, S; Proceedings of the 6th Meeting on Supercritical Fluids, Chemistry and Materials 1999, P187
(13) Weller, S; AIChE J 1956, V2, P59 HCAPLUS
L25 ANSWER 9 OF 23 HCAPLUS COPYRIGHT 2003 ACS
AN 2000:136771 HCAPLUS
DN 132:222678
TI Catalyzed synthesis of DL-.alpha.-tocopherol by phosphomolybdic acid
AU Qian, Dong; Yao, Li-Min; Tan, Yan-Zhi; Cui, Jia
CS School of Chemistry and Chemical Engineering, Central South University of Technology, Changsha, 410083, Peop. Rep. China

- SO Hecheng Huaxue (1999), 7(4), 401-402
CODEN: HEHUE2; ISSN: 1005-1511
- PB Hecheng Huaxue Bianjibu
- DT Journal
- LA Chinese
- CC 30-30 (Terpenes and Terpenoids)
- AB DL-.alpha.-Tocopherol was synthesized by condensing 2,3
5-trimethylhydroquinone with **isophytol** at room temp. using
phosphomolybdic acid (H3PMo12O40) as a catalyst. It was found that
HaPMo12O40 was a high active catalyst for the synthesis of
DL-.alpha.-tocopherol and could easily be sepd. and reused.
- ST tocopherol prepn; **isophytol** condensation catalyst
methylhydroquinone phosphomolybdic acid
- IT Condensation reaction catalysts
(catalyzed synthesis of DL-.alpha.-tocopherol by phosphomolybdic acid)
- IT 12026-57-2, Phosphomolybdic acid (H3PMo12O40)
RL: **CAT (Catalyst use)**; **USES (Uses)**
(catalyzed synthesis of DL-.alpha.-tocopherol by phosphomolybdic acid)
- IT 505-32-8, **Isophytol 700-13-0**, 2,3 5-
Trimethylhydroquinone
RL: **RCT (Reactant)**; **RACT (Reactant or reagent)**
(catalyzed synthesis of DL-.alpha.-tocopherol by phosphomolybdic acid)
- IT **10191-41-0P**, DL-.alpha.-Tocopherol
RL: **SPN (Synthetic preparation)**; **PREP (Preparation)**
(catalyzed synthesis of DL-.alpha.-tocopherol by phosphomolybdic acid)
- L25 ANSWER 10 OF 23 HCAPLUS COPYRIGHT 2003 ACS
- AN 2000:76974 HCAPLUS
- DN 132:122784
- TI Process for the preparation of .alpha.-tocopherol
- IN Hirose, Noriyasu; Inoue, Hiroshi; Matsunami, Toshio; Yoshimura, Takashi;
Morita, Kouzou; Horikawa, Yuh; Iwata, Noriyoshi; Minami, Norio; Hayashi,
Kenji; Seki, Chiaki
- PA Eisai Co., Ltd., Japan
- SO U.S., 7 pp., Cont.-in-part of U.S. 5,886,197.
CODEN: USXXAM
- DT Patent
- LA English
- IC ICM C07D311-20
- NCL 549411000
- CC 30-20 (Terpenes and Terpenoids)
- FAN.CNT 2
- | | PATENT NO. | KIND | DATE | APPLICATION NO. | DATE |
|------|----------------|------|----------|-----------------|----------|
| PI | US 6020505 | A | 20000201 | US 1998-210663 | 19981214 |
| | US 5663376 | A | 19970902 | US 1995-460667 | 19950602 |
| | JP 09188673 | A2 | 19970722 | JP 1997-15371 | 19950727 |
| | JP 09188674 | A2 | 19970722 | JP 1997-15372 | 19950727 |
| | US 5886197 | A | 19990323 | US 1997-826504 | 19970403 |
| PRAI | JP 1994-175167 | A | 19940727 | | |
| | JP 1994-282309 | A | 19941116 | | |
| | US 1995-460667 | A3 | 19950602 | | |
| | US 1997-826504 | A2 | 19970403 | | |
| | JP 1995-191719 | A3 | 19950727 | | |
- OS CASREACT 132:122784; MARPAT 132:122784
- AB A process for the prepn. of .alpha.-tocopherol via cyclocondensation
reaction of trimethylhydroquinone and a specific **phytol** deriv.
or **isophytol** was presented. The cyclocondensation reaction was
conducted in various solvents, such as a carbonate ester, a lower fatty
acid ester R1COOR2 (R1, R2 = alkyl), a mixed solvent of a nonpolar solvent

and an alc., or a mixed solvent of a nonpolar solvent and a lower fatty acid ester. Thus, (.+-.)-.alpha.-tocopherol was prepd in 98.6% yield by cyclocondensation of 2,3,5-trimethylhydroquinone and **isophytol** using hydrochloric acid and zinc chloride as a catalyst and iso-Bu acetate as the solvent. (.+-.)-.alpha.-Tocopherol was similarly prepd in 95.9% using in a hexane and 1-butanol mixed solvent system and in 99.1% yield using hydrobromic acid and zinc bromide in di-Et acetate.

ST tocopherol prepn cyclocondensation

IT Cyclocondensation reaction

(process for the prepn. of .alpha.-tocopherol via cyclocondensation reaction of trimethylhydroquinone a **phytol** deriv. or **isophytol**)

IT Ligroine

RL: NUU (Other use, unclassified); USES (Uses)

(process for the prepn. of .alpha.-tocopherol via cyclocondensation reaction of trimethylhydroquinone a **phytol** deriv. or **isophytol**)

IT 7646-85-7, Zinc chloride, uses 7699-45-8, Zinc bromide

RL: CAT (Catalyst use); USES (Uses)

(process for the prepn. of .alpha.-tocopherol via cyclocondensation reaction of trimethylhydroquinone a **phytol** deriv. or **isophytol**)

IT 10191-41-0P, (.+-.)-.alpha.-Tocopherol

RL: IMF (Industrial manufacture); SPN (Synthetic preparation); PREP (Preparation)

(process for the prepn. of .alpha.-tocopherol via cyclocondensation reaction of trimethylhydroquinone a **phytol** deriv. or **isophytol**)

IT 71-23-8, 1-Propanol, uses 71-36-3, 1-Butanol, uses 71-41-0, 1-Pentanol, uses 75-85-4, tert-Amyl alcohol 96-49-1, Ethylene carbonate 97-62-1, Ethyl isobutyrate 105-54-4, Ethyl butyrate 105-58-8, Diethyl carbonate 108-21-4, 2-Propyl acetate 108-32-7, Propylene carbonate 108-64-5, Ethyl isovalerate 108-88-3, Toluene, uses 109-60-4, 1-Propyl acetate 110-19-0, Isobutyl acetate 110-54-3, Hexane, uses 110-82-7, Cyclohexane, uses 123-86-4, 1-Butyl acetate 142-82-5, Heptane, uses 547-63-7, Methyl isobutyrate 590-01-2 616-38-6, Dimethyl carbonate 623-53-0, Methyl ethyl carbonate 624-24-8, Methyl valerate 638-11-9, Butanoic acid 1-methylethyl ester 3938-95-2, Ethyl pivalate

RL: NUU (Other use, unclassified); USES (Uses)

(process for the prepn. of .alpha.-tocopherol via cyclocondensation reaction of trimethylhydroquinone a **phytol** deriv. or **isophytol**)

IT 505-32-8, **Isophytol** 700-13-0, 2,3,5-

Trimethylhydroquinone 7647-01-0, Hydrochloric acid, reactions 10035-10-6, Hydrobromic acid, reactions

RL: RCT (Reactant); RACT (Reactant or reagent)

(process for the prepn. of .alpha.-tocopherol via cyclocondensation reaction of trimethylhydroquinone a **phytol** deriv. or **isophytol**)

RE.CNT 2 THERE ARE 2 CITED REFERENCES AVAILABLE FOR THIS RECORD

RE

(1) Hirose; US 5663376 1997 HCAPLUS

(2) Hirose; US 5886197 1999 HCAPLUS

L25 ANSWER 11 OF 23 HCAPLUS COPYRIGHT 2003 ACS

AN 2000:34868 HCAPLUS

DN 132:78721

TI Method of producing vitamin E

IN Hyatt, John Anthony

PA Eastman Chemical Company, USA
 SO PCT Int. Appl., 38 pp.
 CODEN: PIXXD2
 DT Patent
 LA English
 IC ICM C07D311-72
 ICS C07D303-16; C07C069-145; C07C043-166
 CC 30-20 (Terpenes and Terpenoids)
 FAN.CNT 3

	PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
PI	WO 2000001686	A1	20000113	WO 1999-US14976	19990701
	W: BR, CN, JP, MX				
	RW: AT, BE, CH, CY, DE, DK, ES, FI, FR, GB, GR, IE, IT, LU, MC, NL, PT, SE				
	WO 2000001685	A1	20000113	WO 1999-US14975	19990701
	W: CN, JP				
	RW: AT, BH, CH, CY, DE, DK, ES, FI, FR, GB, GR, IE, IT, LU, MC, NL, PT, SH				
	BR 9911873	A	20010327	BR 1999-11873	19990701
	EP 1095033	A1	20010502	EP 1999-933650	19990701
	R: AT, BE, CH, DE, DK, ES, FR, GB, GR, IT, LI, LU, NL, SE, MC, PT, IE, SI, LT, LV, FI, RO				
	EP 1095034	A1	20010502	EP 1999-935390	19990701
	R: AT, BE, CH, DE, DK, ES, FR, GB, GR, IT, LI, LU, NL, SE, MC, PT, IE, FI				
	JP 2002519416	T2	20020702	JP 2000-558089	19990701
	JP 2002519417	T2	20020702	JP 2000-558090	19990701
	US 6156913	A	20001205	US 1999-347769	19990706
	US 6242227	B1	20010605	US 1999-352654	19990706
	US 6262279	B1	20010717	US 1999-348192	19990706
	US 6410755	B1	20020625	US 1999-348097	19990706
PRAI	US 1998-91868P	P	19980706		
	US 1998-91983P	P	19980706		
	WO 1999-US14975	W	19990701		
	WO 1999-US14976	W	19990701		
OS	CASREACT 132:78721; MARPAT 132:78721				
AB	<p>The invention provides processes for producing phytol, isophytol, and certain phytol derivs. by a method of oxidizing or epoxidizing geranylgeraniol or geranylgeraniol derivs. to form epoxygeranylgeraniol derivs., reducing or hydrogenating the epoxygeranylgeraniol derivs. to produce epoxyphytol derivs., and then deoxygenating the epoxyphytol derivs. to produce phytol, isophytol, phytene derivs., isophytene derivs., or mixts. thereof. The step of deoxygenating is carried out in the presence of deoxygenation catalysts, including rhenium trioxide compds. The invention also provides methods for the synthesis of certain novel substituted epoxyphytyl compds. and substituted phytene compds. having removable protecting groups. The produced phytol, isophytol, phytene derivs., isophytene derivs., substituted epoxyphytyl compds. and substituted phytene compds. are suitable starting materials for condensation with trimethylhydroquinone derivs. in the presence of Lewis acids to give tocopherol derivs. including .alpha.-tocopherol, vitamin E. Thus, geranylgeraniol is epoxidized using vanadium tris(acetylacetonate) and tert-butylhydroperoxide to give 2,3-epoxygeranylgeraniol in 98% yield. 2,3-Epoxygeranylgeraniol is hydrogenated with Pd/C, and the resulting 2,3-epoxyphytol is deoxygenated with methylrhenium trioxide to give a mixt. of isophytol and phytol in 90% yield.</p>				
ST	vitamin E prepn; tocopherol prepn; phytol prepn tocopherol				

intermediate; **isophytol** prepn tocopherol intermediate
IT Epoxidation
Reduction
(prepn. of **phytol** and **isophytol** as tocopherol intermediates)
IT Tocopherols
RL: SPN (Synthetic preparation); PREP (Preparation)
(prepn. of **phytol** and **isophytol** as tocopherol intermediates)
IT 70197-13-6, Methylrhenium trioxide
RL: **CAT (Catalyst use)**; USES (Uses)
(deoxygenation catalyst; prepn. of **phytol** and **isophytol** as tocopherol intermediates)
IT 13476-99-8, Vanadium tris(acetylacetonate)
RL: **CAT (Catalyst use)**; USES (Uses)
(epoxidn. catalyst; prepn. of **phytol** and **isophytol** as tocopherol intermediates)
IT 7440-05-3, Palladium, uses
RL: **CAT (Catalyst use)**; USES (Uses)
(hydrogenation catalyst; prepn. of **phytol** and **isophytol** as tocopherol intermediates)
IT 505-32-8P, **Isophytol** 106938-96-9P 107438-44-8P
221234-74-8P, 2,3-Epoxygeranylgeraniol 253686-88-3P, dl-**Phytol**
253686-93-0P
RL: IMF (Industrial manufacture); RCT (Reactant); SPN (Synthetic preparation); PREP (Preparation); RACT (Reactant or reagent)
(prepn. of **phytol** and **isophytol** as tocopherol intermediates)
IT 10191-41-0P, dl-.alpha.-Tocopherol
RL: IMF (Industrial manufacture); SPN (Synthetic preparation); PREP (Preparation)
(prepn. of **phytol** and **isophytol** as tocopherol intermediates)
IT 75-91-2, tert-Butylhydroperoxide 80-15-9, Cumene hydroperoxide
700-13-0, Trimethylhydroquinone 7722-84-1, Hydrogen peroxide, reactions 24034-73-9
RL: RCT (Reactant); RACT (Reactant or reagent)
(prepn. of **phytol** and **isophytol** as tocopherol intermediates)
RE.CNT 4 THERE ARE 4 CITED REFERENCES AVAILABLE FOR THIS RECORD
RE
(1) Anon; 1989, 30, P687 HCAPLUS
(2) Eisai; JP 62209072 A 1987 HCAPLUS
(3) Stalla-Bourdillon, B; IND CHIM BEL 1970, V35(1), P13 HCAPLUS
(4) Teijin; DE 2404621 A 1975 HCAPLUS
L25 ANSWER 12 OF 23 HCAPLUS COPYRIGHT 2003 ACS
AN 2000:34593 HCAPLUS
DN 132:78720
TI Manufacture of d,l-.alpha.-tocopherol
IN Aquino, Fabrice; Bonrath, Werner
PA F. Hoffmann-La Roche AG, Switz.
SO Eur. Pat. Appl., 9 pp.
CODEN: EPXXDW
DT Patent
LA English
IC ICM C07D311-72
CC 30-20 (Terpenes and Terpenoids)
FAN.CNT 1
PATENT NO. KIND DATE APPLICATION NO. DATE

PI EP 970953 A1 20000112 EP 1999-112324 19990626
R: AT, BE, CH, DE, DK, ES, FR, GB, GR, IT, LI, LU, NL, SE, MC, PT,
IE, SI, LT, LV, FI, RO
US 6452023 B1 20020917 US 1999-342272 19990629
KR 2000011530 A 20000225 KR 1999-27181 19990707
JP 2000044556 A2 20000215 JP 1999-194076 19990708
BR 9902781 A 20000516 BR 1999-2781 19990708
CN 1241566 A 20000119 CN 1999-110422 19990709
PRAI EP 1998-112842 A 19980710
OS CASREACT 132:78720
AB dl-.alpha.-Tocopherol is prepd. by the condensation of
trimethylhydroquinone and **isophytol** or **phytol** in the
presence of a catalyst that is non-toxic and has no corrosive action.
Thus, 2,3,5-trimethylhydroquinone and **isophytol** are reacted in
the presence of 12-tungstophosphoric acid in hexane to give
dl-.alpha.-tocopherol in 97% yield and 91.4% purity.
ST tocopherol prepn
IT Condensation reaction
(prepn. of dl-.alpha.-tocopherol)
IT Condensation reaction catalysts
Corrosion prevention
(prepn. of dl-.alpha.-tocopherol using a catalyst that is non-toxic and
has no corrosive action)
IT 1343-93-7, 12-Tungstophosphoric acid 12026-57-2, 12-Molybdophosphoric
acid 12027-38-2, 12-Tungstosilicic acid
RL: CAT (Catalyst use); USES (Uses)
(prepn. of dl-.alpha.-tocopherol)
IT 10191-41-0P, dl-.alpha.-Tocopherol
RL: IMF (Industrial manufacture); SPN (Synthetic
preparation); PREP (Preparation)
(prepn. of dl-.alpha.-tocopherol)
IT 150-86-7, **Phytol** 505-32-8, **Isophytol**
700-13-0, 2,3,5-Trimethylhydroquinone
RL: RCT (Reactant); RACT (Reactant or reagent)
(prepn. of dl-.alpha.-tocopherol)
IT 96-49-1, Ethylene carbonate 108-32-7, Propylene carbonate 110-54-3,
Hexane, uses 111-65-9, Octane, uses 142-82-5, Heptane, uses
RL: NUU (Other use, unclassified); USES (Uses)
(solvent; prepn. of dl-.alpha.-tocopherol)
RE.CNT 14 THERE ARE 14 CITED REFERENCES AVAILABLE FOR THIS RECORD
RE
(1) Anon; CN 1123278 A 1996 HCAPLUS
(2) Anon; CA 2154605 A 1996 HCAPLUS
(3) Anon; EP 694541 A 1996 HCAPLUS
(4) Anon; JP 8193079 A 1996
(5) Anon; DE 19603142 A 1997 HCAPLUS
(6) Anon; DE 19617444 A 1997 HCAPLUS
(7) Anon; JP 2644460 B 1997 HCAPLUS
(8) Anon; US 5663376 A 1997 HCAPLUS
(9) Anon; JP 9188673 A 1997
(10) Anon; JP 9188674 A 1997
(11) Anon; WO 9728151 A 1997 HCAPLUS
(12) Anon; EP 0882036 A 1998 HCAPLUS
(13) Anon; CN 1211243 A 1999 HCAPLUS
(14) Anon; US 5886197 A 1999 HCAPLUS

L25 ANSWER 13 OF 23 HCAPLUS COPYRIGHT 2003 ACS
AN 1999:659082 HCAPLUS
DN 131:272041

TI Process for manufacturing D,L-.alpha.-tocopherol in a carbonate solvent
and in the presence of a sulfur-containing acid catalyst
IN Baak, Marcel; Bonrath, Werner; Kreienbuehl, Paul
PA F. Hoffmann-La Roche A.-G., Switz.
SO Eur. Pat. Appl., 8 pp.
CODEN: EPXXDW

DT Patent

LA English

IC ICM C07D311-72

CC 30-20 (Terpenes and Terpenoids)

FAN.CNT 1

	PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
PI	EP 949255	A1	19991013	EP 1999-106507	19990330
	R: AT, BE, CH, DE, DK, ES, FR, GB, GR, IT, LI, LU, NL, SE, MC, PT, IE, SI, LT, LV, FI, RO				
	US 6066745	A	20000523	US 1999-282411	19990331
	CN 1234402	A	19991110	CN 1999-104919	19990406
	JP 2000053673	A2	20000222	JP 1999-98266	19990406
PRAI	EP 1998-106237	A	19980406		
OS	CASREACT 131:272041				
AB	A process for the manuf. of D,L-.alpha.-tocopherol by acid-catalyzed condensation of trimethylhydroquinone with isophytol comprises carrying out the condensation in the presence of sulfuric acid, methanesulfonic acid, ethanesulfonic acid, trifluoromethanesulfonic acid, p-toluenesulfonic acid, or fluorosulfonic acid (in concn. of .ltoreq. 0.4 wt.% based on the wt. of isophytol) in ethylene or propylene carbonate or a mixt. of both carbonates, or in a mixt. of one or both of the carbonates and a non-polar solvent. Thus, 2,3,5-trimethylhydroquinone was condensed with isophytol in ethylene carbonate-heptane contg. 10% vol./vol. H2SO4 to give 94.1% D,L-.alpha.-tocopherol.				
ST	tocopherol prepn; methylhydroquinone cycloaddn isophytol ; catalyst acid methylhydroquinone cycloaddn isophytol ; solvent carbonate methylhydroquinone cycloaddn isophytol				
IT	Cycloaddition reaction catalysts (acids; process for manufg. D,L-tocopherol in a carbonate solvent and in presence of a sulfur-contg. acid catalyst)				
IT	Carbonates, uses RL: NUU (Other use, unclassified); USES (Uses) (as solvents; for cycloaddn. reaction of trimethylhydroquinone with isophytol in prepn. of .alpha.-tocopherol)				
IT	Solvents (carbonates; for cycloaddn. reaction of trimethylhydroquinone with isophytol in prepn. of .alpha.-tocopherol)				
IT	Cycloaddition reaction (process for manufg. D,L-tocopherol in a carbonate solvent and in presence of a sulfur-contg. acid catalyst)				
IT	Acids, uses RL: CAT (Catalyst use) ; USES (Uses) (process for manufg. D,L-tocopherol in a carbonate solvent and in presence of a sulfur-contg. acid catalyst)				
IT	75-75-2, Methanesulfonic acid 104-15-4, p-Toluenesulfonic acid, uses 594-45-6, Ethanesulfonic acid 1493-13-6, Trifluoromethanesulfonic acid 7664-93-9, Sulfuric acid, uses 7789-21-1, Fluorosulfonic acid RL: CAT (Catalyst use) ; USES (Uses) (process for manufg. D,L-tocopherol in a carbonate solvent and in presence of a sulfur-contg. acid catalyst)				
IT	96-49-1, Ethylene carbonate 108-32-7, Propylene carbonate RL: NUU (Other use, unclassified); USES (Uses) (process for manufg. D,L-tocopherol in a carbonate solvent and in				

presence of a sulfur-contg. acid catalyst)
IT 505-32-8, **Isophytol 700-13-0**, Trimethylhydroquinone
RL: RCT (Reactant); RACT (Reactant or reagent)
(process for manufg. D,L-tocopherol in a carbonate solvent and in
presence of a sulfur-contg. acid catalyst)
IT 10191-41-0P, (.+-.)-.alpha.-Tocopherol
RL: SPN (Synthetic preparation); PREP (Preparation)
(process for manufg. D,L-tocopherol in a carbonate solvent and in
presence of a sulfur-contg. acid catalyst)
RE.CNT 5 THERE ARE 5 CITED REFERENCES AVAILABLE FOR THIS RECORD
RE
(1) Basf Aktiengesellschaft; WO 9728151 A 1997 HCAPLUS
(2) Eisai Co Ltd; EP 0694541 A 1996 HCAPLUS
(3) Greenbaum, S; US 3708505 A 1973 HCAPLUS
(4) Kajiwara, M; HETEROCYCLES 1981, V15(2), P1209 HCAPLUS
(5) Urano, S; HETEROCYCLES 1984, V22(9), P1975 HCAPLUS
L25 ANSWER 14 OF 23 HCAPLUS COPYRIGHT 2003 ACS
AN 1999:119233 HCAPLUS
DN 130:282189
TI Synthesis of D,L-.alpha.-Tocopherol Using Strong Solid Acids as Catalysts
AU Schager, Frank; Bonrath, Werner
CS Vitamin Research and Technology Development, F. Hoffmann-La Roche Ltd.,
Basel, CH-4070, Switz.
SO Journal of Catalysis (1999), 182(1), 282-284
CODEN: JCTLA5; ISSN: 0021-9517
PB Academic Press
DT Journal
LA English
CC 30-20 (Terpenes and Terpenoids)
OS CASREACT 130:282189
AB The synthesis of DL-.alpha.-tocopherol starting from trimethylhydroquinone
and **isophytol** using a heterogeneous solid acid catalyst, e.g.,
Nafion NR 50, is described. Advantages of this new procedure are high
yield and selectivity, facile recovery of the catalyst, no waste problems,
and mild reaction conditions. (c) 1999 Academic Press.
ST tocopherol synthesis solid acid catalyst Friedel Craft reaction
IT Friedel-Crafts reaction
Friedel-Crafts reaction catalysts
(synthesis of DL-.alpha.-tocopherol using strong solid acid catalysts)
IT Acids, uses
RL: CAT (Catalyst use); USES (Uses)
(synthesis of DL-.alpha.-tocopherol using strong solid acid catalysts)
IT 96-22-0, Diethyl ketone 96-48-0, .gamma.-Butyrolactone 108-10-1,
Isobutyl methyl ketone 108-32-7, Propylene carbonate 108-88-3,
Toluene, uses
RL: NUU (Other use, unclassified); USES (Uses)
(solvent; synthesis of DL-.alpha.-tocopherol using strong solid acid
catalysts)
IT 9037-24-5, Amberlyst 15 118473-68-0, Nafion NR 50
RL: CAT (Catalyst use); USES (Uses)
(synthesis of DL-.alpha.-tocopherol using strong solid acid catalysts)
IT 505-32-8, **Isophytol 700-13-0**
RL: RCT (Reactant); RACT (Reactant or reagent)
(synthesis of DL-.alpha.-tocopherol using strong solid acid catalysts)
IT 10191-41-0P, DL-.alpha.-Tocopherol
RL: SPN (Synthetic preparation); PREP (Preparation)
(synthesis of DL-.alpha.-tocopherol using strong solid acid catalysts)
RE.CNT 18 THERE ARE 18 CITED REFERENCES AVAILABLE FOR THIS RECORD
RE

- (1) Bonrath, W; EP 782993 1997 HCAPLUS
- (2) Childs, R; J Org Chem 1982, V47, P4207
- (3) Finnan, J; EP 100471 1982 HCAPLUS
- (4) Greenbaum, S; US 3708505 1971 HCAPLUS
- (5) Harmer, M; J Am Chem Soc 1996, V118, P7708 HCAPLUS
- (6) Heidekum, A; J Catal 1998, V176, P260 HCAPLUS
- (7) Isler, O; Vitamine I 1988, P126
- (8) Karrer, P; US 2411967 1946 HCAPLUS
- (9) Matsui, M; Bull Chem Soc Jpn 1995, V68, P3569 HCAPLUS
- (10) Nelan, D; US 3444213 1969 HCAPLUS
- (11) Olah, G; J Org Chem 1977, V42, P4187 HCAPLUS
- (12) Olah, G; Science 1979, V206, P13 HCAPLUS
- (13) Olah, G; Synthesis 1978, P672 HCAPLUS
- (14) Olah, G; Synthesis 1986, P513 HCAPLUS
- (15) Schulz, B; DE 3203487 1983 HCAPLUS
- (16) Skoda, A; CS 254402 1989
- (17) Tachibana, Y; Bull Chem Soc Jpn 1977, V50, P2477 HCAPLUS
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L25 ANSWER 15 OF 23 HCAPLUS COPYRIGHT 2003 ACS

AN 1998:341555 HCAPLUS

DN 129:28106

TI Process for manufacturing d,l-.alpha.-tocopherol via catalyzed condensation

IN Baak, Marcel; ~~Bonrath~~, Werner; Pauling, Horst

PA F. Hoffmann-La Roche A.-G., Switz.

SO PCT Int. Appl., 16 pp.

CODEN: PIXXD2

DT Patent

LA English

IC ICM C07D311-00

CC 30-20 (Terpenes and Terpenoids)

FAN.CNT 1

	PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
PI	WO 9821197	A2	19980522	WO 1997-EP6227	19971110
	W: CN, JP, KR				
	RW: AT, BE, CH, DE, DK, ES, FI, FR, GB, GR, IE, IT, LU, MC, NL, PT, SE				
	US 5908939	A	19990601	US 1997-951273	19971016
	EP 937055	A2	19990825	EP 1997-950150	19971110
	EP 937055	B1	20020403		
	R: BE, CH, DE, DK, ES, FR, GB, IT, LI, NL, SE, IE				
	CN 1237163	A	19991201	CN 1997-199633	19971110
	JP 2001504111	T2	20010327	JP 1998-522153	19971110
	ES 2173500	T3	20021016	ES 1997-950150	19971110
	KR 2000053181	A	20000825	KR 1999-704139	19990510
PRAI	EP 1996-118037	A	19961111		
	WO 1997-EP6227	W	19971110		

OS CASREACT 129:28106

AB A process for the manuf. of dl-.alpha.-tocopherol by the catalyzed condensation of trimethylhydroquinone with **isophytol** is described. The method comprises carrying out the condensation in the presence of bis-(trifluoromethylsulfonyl)amine [HN(SO₂CF₃)₂] or a metal salt thereof, of the formula M[N(SO₂CF₃)₂]_n (I) [M = metal atom selected from the group of lithium, boron, magnesium, aluminum, silicon, scandium, titanium, vanadium, manganese, iron, cobalt, nickel, copper, zinc, yttrium, zirconium, rhodium, palladium, silver, tin, lanthanum, cerium, neodymium, praseodymium, europium, dysprosium, ytterbium, hafnium, platinum and gold; n = the corresponding valency (1, 2, 3 or 4) of the metal atom M], as the catalyst, or of a combination of a metal salt of

formula I and a strong Bronsted acid as the catalyst system in an org. solvent. Thus, a mixt. of trimethylhydroquinone, **isophytol** and bis-(trifluoromethylsulfonyl)amine in toluene are boiled at 140.degree.C to give dl-.alpha.-tocopherol in 90% yield.

ST tocopherol prepn catalyzed condensation

IT Condensation reaction

Condensation reaction catalysts

(prepn. of dl-.alpha.-tocopherol via catalyzed condensation)

IT 7440-16-6D, Rhodium, bis(trifluoromethylsulfonyl)amide complexes, uses 82113-65-3, Bis-(trifluoromethylsulfonyl)amine 82113-65-3D, rhodium complexes 90076-65-6, Lithium bis(trifluoromethylsulfonyl)amide 133395-16-1 162715-14-2 168106-25-0 168106-26-1 175438-45-6 176726-07-1 189114-61-2 189114-76-9 189114-86-1 207861-54-9 207861-55-0 207861-57-2 207861-59-4 207861-61-8 207861-63-0 207861-64-1 207861-65-2 207861-66-3 207861-67-4 207861-68-5 207861-69-6 207861-70-9 207861-71-0 207861-72-1

RL: CAT (Catalyst use); USES (Uses)

(prepn. of dl-.alpha.-tocopherol via catalyzed condensation)

IT 10191-41-0P, dl-.alpha.-Tocopherol

RL: IMF (Industrial manufacture); SPN (Synthetic preparation); PREP (Preparation)

(prepn. of dl-.alpha.-tocopherol via catalyzed condensation)

IT 505-32-8, **Isophytol** 700-13-0, Trimethylhydroquinone

RL: RCT (Reactant); RACT (Reactant or reagent)

(prepn. of dl-.alpha.-tocopherol via catalyzed condensation)

L25 ANSWER 16 OF 23 HCAPLUS COPYRIGHT 2003 ACS

AN 1998:25397 HCAPLUS

DN 128:154258

TI Preparation of .alpha.-tocopherol with scandium complex as recyclable catalyst

IN Yamamoto, Takashi

PA Eisai Co., Ltd., Japan; Central Glass Co., Ltd.

SO Jpn. Kokai Tokkyo Koho, 5 pp.

CODEN: JKXXAF

DT Patent

LA Japanese

IC ICM C07D311-72

ICS B01J031-22; C07B061-00

CC 30-20 (Terpenes and Terpenoids)

FAN.CNT 1

	PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
PI	JP 10001477	A2	19980106	JP 1996-171638	19960612
PRAI	JP 1996-171638		19960612		

OS CASREACT 128:154258

AB .alpha.-Tocopherol (I) is prepd. by condensation of trimethylhydroquinone (II) with **phytol**, phytyl halides, phytyl acetate, phytyl methanesulfonate, phytyl ethanesulfonate, phytyl benzenesulfonate, phytyl toluenesulfonate, or **isophytol** in the presence of Sc[N(CF₃SO₂)₂]₃. II was refluxed with **isophytol** and Sc[N(CF₃SO₂)₂]₃ for 2.5 h in the dark to give 93% I.

ST alpha tocopherol prepn scandium complex catalyst; Friedel Crafts reaction methylhydroquinone **phytol isophytol**

IT Cyclocondensation reaction catalysts

Friedel-Crafts reaction catalysts

(prepn. of .alpha.-tocopherol with scandium complex as recyclable catalyst)

IT 144026-79-9, Scandium triflate

RL: CAT (Catalyst use); USES (Uses)

(prepn. of .alpha.-tocopherol with scandium complex as recyclable catalyst)

IT 10191-41-0P

RL: IMF (Industrial manufacture); SPN (Synthetic preparation); PREP (Preparation)

(prepn. of .alpha.-tocopherol with scandium complex as recyclable catalyst)

IT 150-86-7, **Phytol** 505-32-8, **Isophytol**

700-13-0, Trimethylhydroquinone 10236-16-5, Phytol acetate
202747-36-2 202747-37-3 202747-38-4 202747-39-5

RL: RCT (Reactant); RACT (Reactant or reagent)

(prepn. of .alpha.-tocopherol with scandium complex as recyclable catalyst)

L25 ANSWER 17 OF 23 HCAPLUS COPYRIGHT 2003 ACS

AN 1997:591002 HCAPLUS

DN 127:176579

TI Preparation of dl-.alpha.-tocopherol and its acetate via reaction of 2,3,5-trimethylhydroquinone with **phytol** or **isophytol**

IN Jaedicke, Hagen; Grafen, Paul; Laas, Harald

PA BASF A.-G., Germany

SO Ger. Offen., 5 pp.

CODEN: GWXXBX

DT Patent

LA German

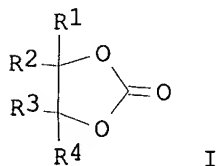
IC ICM C07D311-70

ICS C07D317-36

CC 30-20 (Terpenes and Terpenoids)

FAN.CNT 2

	PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
PI	DE 19603142	A1	19970731	DE 1996-19603142	19960129
	WO 9728151	A1	19970807	WO 1997-EP324	19970124
	W: CN, JP, RU, SK, US				
	RW: AT, BE, CH, DE, DK, ES, FI, FR, GB, GR, IE, IT, LU, MC, NL, PT, SE				
	EP 882036	A1	19981209	EP 1997-901601	19970124
	R: CH, DE, FR, GB, LI				
	CN 1211243	A	19990317	CN 1997-191917	19970124
	RU 2160258	C2	20001210	RU 1998-116519	19970124
	JP 2002502360	T2	20020122	JP 1997-527279	19970124
PRAI	DE 1996-19603142	A	19960129		
	DE 1996-19617444	A	19960502		
	WO 1997-EP324	W	19970124		
OS	MARPAT 127:176579				
GI					



AB A procedure for the prepn. of dl-.alpha.-tocopherol and its acetate via acid-catalyzed reaction of 2,3,5-trimethylhydroquinone with **phytol** or **isophytol** in a soln. at elevated temp. and subsequent esterification of tocopherol with acetic anhydride is marked in that the

reaction takes place in a cyclic carbonate I [R1, R2, R3 = H, Me, Et; R4 = H, (un)substituted Ph, alkoxy] at 100 - 200.degree.. Thus, 2,3,5-trimethylhydroquinone in propylene carbonate contg. catalytic orthoboric and oxalic acids at 150.degree. is treated with a soln. of **isophytol** in heptane over 10 mins. to give 95% dl-.alpha.-tocopherol.

- ST tocopherol acetate prepn; trimethylhydroquinone reaction **phytol**
isophytol
- IT 77-92-9, Citric acid, uses 87-69-4, (+)-L-Tartaric acid, uses 109-63-7, Boron trifluoride etherate 144-62-7, Oxalic acid, uses 10043-35-3, Orthoboric acid, uses
RL: CAT (Catalyst use); USES (Uses)
(prepn. of .alpha.-tocopherol and its acetate via reaction of trimethylhydroquinone with **phytol** or **isophytol**)
- IT 96-49-1, Ethylene carbonate 108-32-7, Propylene carbonate 4437-69-8, Isobutylene carbonate 4437-85-8, 1,2-Butylene carbonate
RL: NUU (Other use, unclassified); USES (Uses)
(prepn. of .alpha.-tocopherol and its acetate via reaction of trimethylhydroquinone with **phytol** or **isophytol**)
- IT 108-24-7, Acetic anhydride 150-86-7, **Phytol** 505-32-8, **Isophytol** 700-13-0, 2,3,5-Trimethylhydroquinone
RL: RCT (Reactant); RACT (Reactant or reagent)
(prepn. of .alpha.-tocopherol and its acetate via reaction of trimethylhydroquinone with **phytol** or **isophytol**)
- IT 10191-41-0P, dl-.alpha.-Tocopherol
RL: RCT (Reactant); SPN (Synthetic preparation); PREP (Preparation); RACT (Reactant or reagent)
(prepn. of .alpha.-tocopherol and its acetate via reaction of trimethylhydroquinone with **phytol** or **isophytol**)
- IT 52225-20-4P, dl-.alpha.-Tocopherol acetate
RL: SPN (Synthetic preparation); PREP (Preparation)
(prepn. of .alpha.-tocopherol and its acetate via reaction of trimethylhydroquinone with **phytol** or **isophytol**)

L25 ANSWER 18 OF 23 HCAPLUS COPYRIGHT 2003 ACS

AN 1997:552632 HCAPLUS

DN 127:149274

TI Use of hydrogen bis(oxalato)borate as a condensation reaction catalyst

IN Fuerbringer, Claude

PA F. Hoffmann-La Roche Ag, Switz.

SO Eur. Pat. Appl., 5 pp.

CODEN: EPXXDW

DT Patent

LA French

IC ICM C07B037-00

ICS C07C041-48; C07C067-293; C07C403-10; C07D311-72

CC 30-20 (Terpenes and Terpenoids)

FAN.CNT 2

	PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
PI	EP 784042	A1	19970716	EP 1997-100109	19970107
	R: AT, BE, CH, DE, DK, ES, FR, GB, IT, LI, NL				
	CN 1161251	A	19971008	CN 1997-102008	19970108
	JP 09192493	A2	19970729	JP 1997-1788	19970109
PRAI	CH 1996-96	A	19960112		

OS CASREACT 127:149274

AB Hydrogen bis(oxalato)borate (I) is used as a catalyst in condensation reactions, such as Friedel-Crafts reactions, vinyl ether condensations, acylations, esp. in the prepn. of dihydro-vitamin K1, d,l-.alpha.-tocopherol and its acetate. Thus, trimethylhydroquinone is condensed with

isophytol in presence of I to give 92% d,l-.alpha.-tocopherol, which is acetylated in 92% yield in presence of I.

ST hydrogen bisoxalatoborate prepn condensation reaction catalyst; tocopherol prepn hydrogen bisoxalatoborate catalyst

IT Condensation reaction catalysts
(use of hydrogen bis(oxalato)borate as a condensation reaction catalyst)

IT 83145-82-8P
RL: **CAT (Catalyst use)**; SPN (Synthetic preparation); PREP (Preparation); USES (Uses)
(use of hydrogen bis(oxalato)borate as a condensation reaction catalyst)

IT 144-62-7, Ethanedioic acid, reactions 505-32-8, **Isophytol**
534-15-6, Acetaldehyde dimethylacetal 700-13-0,
Trimethylhydroquinone 1303-86-2, Diboron trioxide, reactions 2211-28-1
6068-62-8 7319-16-6, Methyl propenyl ether 16910-82-0
RL: RCT (Reactant); RACT (Reactant or reagent)
(use of hydrogen bis(oxalato)borate as a condensation reaction catalyst)

IT **10191-41-0P**, dl-.alpha.-Tocopherol
RL: RCT (Reactant); SPN (**Synthetic preparation**); PREP (**Preparation**); RACT (Reactant or reagent)
(use of hydrogen bis(oxalato)borate as a condensation reaction catalyst)

IT 52225-20-4P, dl-.alpha.-Tocopherol acetate 98944-42-4P 103325-26-4P
193347-01-2P 193347-05-6P
RL: SPN (Synthetic preparation); PREP (Preparation)
(use of hydrogen bis(oxalato)borate as a condensation reaction catalyst)

L25 ANSWER 19 OF 23 HCAPLUS COPYRIGHT 2003 ACS

AN 1997:533639 HCAPLUS

DN 127:190871

TI Method of producing (.+-.)-tocopherol or (.+-.)-tocopheryl acetate

IN Jaedicke, Hagen; Grafen, Paul; Laas, Harald

PA Basf A.-G., Germany; Jaedicke, Hagen; Grafen, Paul; Laas, Harald

SO PCT Int. Appl., 21 pp.

CODEN: PIXXD2

DT Patent

LA German

IC ICM C07D311-72

CC 30-30 (Terpenes and Terpenoids)

Section cross-reference(s): 45

FAN.CNT 2

	PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
PI	WO 9728151	A1	19970807	WO 1997-EP324	19970124
	W: CN, JP, RU, SK, US				
	RW: AT, BE, CH, DE, DK, ES, FI, FR, GB, GR, IE, IT, LU, MC, NL, PT, SE				
	DE 19603142	A1	19970731	DE 1996-19603142	19960129
	DE 19617444	A1	19971106	DE 1996-19617444	19960502
	EP 882036	A1	19981209	EP 1997-901601	19970124
	R: CH, DE, FR, GB, LI				
	RU 2160258	C2	20001210	RU 1998-116519	19970124
	JP 2002502360	T2	20020122	JP 1997-527279	19970124
PRAI	DE 1996-19603142	A	19960129		
	DE 1996-19617444	A	19960502		
	WO 1997-EP324	W	19970124		
OS	CASREACT 127:190871				
AB	A method is disclosed of producing (.+-.)-.alpha.-tocopherol or				

(.+-.)-.alpha.-tocopheryl acetate by acid catalyzed reaction of 2,3,5-trimethylhydroquinone (TMH) with **phytol** or **isophytol** (IP) in a solvent at raised temp. and, where appropriate, subsequent esterification of the tocopherol thus obtained with acetic anhydride. The method is characterized in that the reaction is carried out in an optionally substituted cyclic five-ring carbonate such as 1,2-propylene carbonate, or in an optionally substituted five-ring lactone such as .gamma.-butyrolactone, as the solvent at temps. of between 50 and 200.degree.. The reaction works particularly well if, following the reaction of TMH and **phytol** or IP, the tocopherol which separates out as an upper phase as the reaction mixt. cools and/or the reaction mixt. is extd. using a suitable aliph. hydrocarbon, the tocopherol is isolated by distn. from the ext., and the sepd. cyclic carbonate or .gamma.-lactone which can contain excess TMH and acidic catalyst is reused as solvent. In many cases, it has been shown to be advantageous to carry out the reaction of TMH with **phytol** or IP with removal of the water as azeotrope, using a suitable hydrocarbon and/or in the presence of a mixt. of ortho-boric acid with oxalic acid, tartaric acid or citric acid, or alternatively in the presence of BF3 etherate as the acidic catalyst. Surprisingly, the five-ring carbonates and five-ring lactones prove to have sufficient stability under the reaction conditions and suitable solvent characteristics to permit the process to be carried out continuously.

ST tocopherol acetate prepn
 IT 77-92-9, Citric acid, uses 87-69-4, Tartaric acid, uses 144-62-7, Oxalic acid, uses 10043-35-3, Ortho-boric acid, uses
 RL: CAT (Catalyst use); USES (Uses)
 (method of producing (.+-.)-tocopherol or (.+-.)-tocopheryl acetate)
 IT 10191-41-0P, (.+-.)-.alpha.-Tocopherol 52225-20-4P,
 (.+-.)-.alpha.-Tocopheryl acetate
 RL: IMF (Industrial manufacture); SPN (Synthetic preparation); PREP (Preparation)
 (method of producing (.+-.)-tocopherol or (.+-.)-tocopheryl acetate)
 IT 96-48-0, .gamma.-Butyrolactone 108-32-7, 1,2-Propylene carbonate
 RL: MSC (Miscellaneous)
 (method of producing (.+-.)-tocopherol or (.+-.)-tocopheryl acetate)
 IT 150-86-7, **Phytol** 505-32-8, **Isophytol** 700-13-0, 2,3,5-Trimethylhydroquinone
 RL: RCT (Reactant); RACT (Reactant or reagent)
 (method of producing (.+-.)-tocopherol or (.+-.)-tocopheryl acetate)

L25 ANSWER 20 OF 23 HCAPLUS COPYRIGHT 2003 ACS
 AN 1997:496858 HCAPLUS
 DN 127:109079
 TI Process for the preparation of d,l-.alpha.-tocopherol in the presence of a perfluoroalkenesulfonic acid catalyst
 IN Bonrath, Werner
 PA F. Hoffmann-La Roche Ag, Switz.
 SO Eur. Pat. Appl., 5 pp.
 CODEN: EPXXDW
 DT Patent
 LA German
 IC ICM C07D311-72
 CC 30-20 (Terpenes and Terpenoids)
 FAN.CNT 1

	PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
PI	EP 782993	A1	19970709	EP 1996-120823	19961223
	R: AT, BE, CH, DE, DK, ES, FR, GB, IT, LI, NL				
	JP 09194472	A2	19970729	JP 1996-346857	19961226

CN 1160049 A 19970924 CN 1996-123196 19961227
 US 5900494 A 19990504 US 1997-778479 19970103
 PRAI CH 1996-31 19960105
 OS CASREACT 127:109079
 AB The title process involves reacting trimethylhydroquinone with **isophytol** in the presence of a polyperfluoroalkenesulfonic acid in an aprotic solvent between 80 and 140.degree.. Thus, a mixt. of trimethylhydroquinone and Nafion NR 50 in toluene was heated at 107.degree., **isophytol** was added over 2 h, and the resulting mixt. was refluxed for 30 min to give 75.33% (.+-.)-.alpha.-tocopherol.
 ST tocopherol prepn; methylhydroquinone condensation **isophytol**;
 IT condensation trimethylhydroquinone **isophytol**
 IT Sulfonic acids, uses
 RL: **CAT (Catalyst use); USES (Uses)**
 (alkene, polyperfluoro; prepn. of .alpha.-tocopherol in presence of a perfluoroalkenesulfonic acid catalyst)
 IT Condensation reaction
 Condensation reaction catalysts
 Solvents
 (prepn. of .alpha.-tocopherol in presence of a perfluoroalkenesulfonic acid catalyst)
 IT 118473-68-0, Nafion NR 50
 RL: **CAT (Catalyst use); USES (Uses)**
 (prepn. of .alpha.-tocopherol in presence of a perfluoroalkenesulfonic acid catalyst)
 IT **10191-41-0P, Ephanyl**
 RL: **IMF (Industrial manufacture); SPN (Synthetic preparation); PREP (Preparation)**
 (prepn. of .alpha.-tocopherol in presence of a perfluoroalkenesulfonic acid catalyst)
 IT 60-29-7, Diethyl ether, uses 96-48-0, .gamma.-Butyrolactone 108-88-3, Toluene, uses 141-78-6, Ethyl acetate, uses
 RL: **NUU (Other use, unclassified); USES (Uses)**
 (prepn. of .alpha.-tocopherol in presence of a perfluoroalkenesulfonic acid catalyst)
 IT 505-32-8, **Isophytol 700-13-0**, Trimethylhydroquinone
 RL: **RCT (Reactant); RACT (Reactant or reagent)**
 (prepn. of .alpha.-tocopherol in presence of a perfluoroalkenesulfonic acid catalyst)
 L25 ANSWER 21 OF 23 HCAPLUS COPYRIGHT 2003 ACS
 AN 1983:72494 HCAPLUS
 DN 98:72494
 TI .alpha.-Tocopherol
 PA Kuraray Co., Ltd., Japan
 SO Jpn. Tokkyo Koho, 2 pp.
 CODEN: JAXXAD
 DT Patent
 LA Japanese
 IC C07D311-72
 CC 30-20 (Terpenes and Terpenoids)
 FAN.CNT 1

	PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
PI	JP 57040150	B4	19820825	JP 1979-75511	19790614
	JP 55027174	A2	19800227		
PRAI	JP 1979-75511		19790614		

 AB .alpha.-Tocopherol (I) was prepd. by condensation of trimethylhydroquinone (II) with phytyl halide in the presence of Sn. Thus, phytyl chloride, prepd. from 28.5 g **isophytol**, was added to a refluxing mixt. of

12.17 g II, 1.0 g Sn, and 20 mL ligroin over 3 h, the mixt. refluxed for 3 h, 16.33 g Ac₂O and 3.27 g NaOAc were added, and the resulting mixt. was refluxed 2 h to give 38.5 g (+-)-.alpha.-tocopherol acetate, which was hydrolyzed to I. Use of 4.5 g Fe instead of Sn gave only 20.16 g (+-)-.alpha.-tocopherol acetate.

ST alpha tocopherol; condensation trimethylhydroquinone isophytol chloride; tin catalyst condensation tocopherol

IT Condensation reaction
(of trimethylhydroquinone with phytol chloride in .alpha.-tocopherol prepn.)

IT Condensation reaction catalysts
(tin, for trimethylhydroquinone and phytol chloride, in tocopherol prepn.)

IT 7440-31-5, uses and miscellaneous
RL: **CAT (Catalyst use)**; **USES (Uses)**
(catalyst, for condensation of trimethylhydroquinone and phytol chloride, in tocopherol prepn.)

IT **700-13-0**
RL: **RCT (Reactant)**; **RACT (Reactant or reagent)**
(condensation of, with phytol chloride)

IT 84416-26-2
RL: **RCT (Reactant)**; **RACT (Reactant or reagent)**
(condensation of, with trimethylhydroquinone in prepn. of .alpha.-tocopherol)

IT 7695-91-2P **10191-41-0P**
RL: **SPN (Synthetic preparation)**; **PREP (Preparation)**
(prepn. of, from trimethylhydroquinone and phytol chloride)

L25 ANSWER 22 OF 23 HCAPLUS COPYRIGHT 2003 ACS

AN 1978:443854 HCAPLUS

DN 89:43854

TI dl-.alpha.-Tocopherol

IN Yoshino, Youzиро; Kondo, Kazuko

PA Nisshin Flour Milling Co., Ltd., Japan

SO Ger. Offen., 10 pp.

CODEN: GWXXBX

DT Patent

LA German

IC C07D311-72

CC 30-30 (Terpenoids)

Section cross-reference(s): 27

FAN.CNT 1

	PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
PI	DE 2743920	A1	19780330	DE 1977-2743920	19770929
	DE 2743920	C2	19841018		
	JP 53044570	A2	19780421	JP 1976-115965	19760929
	JP 60019309	B4	19850515		
	US 4217285	A	19800812	US 1979-11882	19790213
PRAI	JP 1976-115965		19760929		
	US 1977-837640		19770928		

AB DL-.alpha.-Tocopherol (I) was prepd. with high purity by condensing trimethylhydroquinone (II) with **phytol** or **isophytol** in the presence of silicic acid-Al₂O₃ and/or silica gel, ZnCl₂, and a protonic acid. Thus, a mixt. of 25.0 g **isophytol**, 13.5 g II, 13.5 g silicic acid-Al₂O₃ or -silica gel, 6.0 g ZnCl₂, and 0.5 g 35% HCl was heated at 68.degree. for 4 h to give 99.8% I (95.8% purity).

ST alpha tocopherol

IT Silica gel, uses and miscellaneous

RL: **CAT (Catalyst use)**; **USES (Uses)**

- (catalyst from silicic acid, zinc chloride, acid, and, for condensation of trimethylhydroquinone with **phytol**)
- IT 1343-98-2
RL: CAT (Catalyst use); USES (Uses)
(catalyst from aluminum oxide, zinc chloride, acid, and, for condensation of trimethylhydroquinone with **phytol**)
- IT 7646-85-7, uses and miscellaneous
RL: CAT (Catalyst use); USES (Uses)
(catalysts from silicic acid, aluminum oxide, acid, and, for condensation of trimethylhydroquinone with **phytol**)
- IT 7647-01-0, uses and miscellaneous 7664-38-2, uses and miscellaneous
7664-93-9, uses and miscellaneous
RL: CAT (Catalyst use); USES (Uses)
(catalysts from silicic acid, aluminum oxide, zinc chloride and, for condensation of trimethylhydroquinone with **phytol**)
- IT 53809-96-4, uses and miscellaneous
RL: CAT (Catalyst use); USES (Uses)
(catalysts from silicic acid, zinc chloride, acid, and, for condensation of trimethylhydroquinone with **phytol**)
- IT 700-13-0
RL: RCT (Reactant); RACT (Reactant or reagent)
(condensation of, with **phytol**)
- IT 150-86-7 505-32-8
RL: RCT (Reactant); RACT (Reactant or reagent)
(condensation of, with trimethylhydroquinone)
- IT 10191-41-0P
RL: SPN (Synthetic preparation); PREP (Preparation)
(prepn. of, by condensation of trimethylhydroquinone with **phytol**)

L25 ANSWER 23 OF 23 HCAPLUS COPYRIGHT 2003 ACS

AN 1975:16963 HCAPLUS

DN 82:16963

TI .alpha.-Tocopherol

IN Omura, Yoshiaki; Nishida, Takashi; Tanomura, Masahisa; Tamai, Yoshin; Mori, Fumio; Hosogai, Takeo; Ninagawa, Yoichi; Itoi, Kazuo

PA Kuraray Co., Ltd.

SO Jpn. Kokai Tokkyo Koho, 3 pp.

CODEN: JKXXAF

DT Patent

LA Japanese

NCL 16E41

CC 30-40 (Terpenoids)

FAN.CNT 1

	PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
PI	JP 49070972	A2	19740709	JP 1972-114039	19721113
	JP 57038588	B4	19820816		
PRAI	JP 1972-114039		19721113		

AB .alpha.-Tocopherol (I) was prepd. by condensing **isophytol** or **phytol** (II) with trimethylhydroquinone (III) in the presence of catalysts comprising Friedel-Craft catalyst, Zn (or Sn), and traces of acids. E.g., 29.65 g II was added to a mixt. of III 15.22, BF₃.Et₂O 14.19, Zn 1.31, and AcOH 1 g in hexane in 2 hr with reflux and the whole kept refluxed 2 hr with azeotropic removal of resultant H₂O to give 38.33 g dl-I.

ST tocopherol; **phytol** condensation trimethylhydroquinone catalyst; hydroquinone trimethyl condensation **phytol**; catalyst condensation **phytol** trimethylhydroquinone

IT Condensation reaction catalysts

(boron trifluoride, for **phytol** with trimethylhydroquinone)

IT 109-63-7
RL: CAT (Catalyst use); USES (Uses)
(catalysts, for condensation of **phytol** with
trimethylhydroquinone)

IT 700-13-0
RL: RCT (Reactant); RACT (Reactant or reagent)
(condensation of, with **phytol**, catalysts for)

IT 150-86-7
RL: RCT (Reactant); RACT (Reactant or reagent)
(condensation of, with trimethylhydroquinone, catalysts for)

IT 10191-41-0P
RL: SPN (Synthetic preparation); PREP (Preparation)
(prepn. of)



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